NiX Thermo-chemical Treatment of Animal Manure for Biogas Production

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1 Executive summary

The thermo-chemical pre-treatment method, NiX, was optimised with respect to pressure, temperature and base addition, and tested on different types of biomass in batch (part 1) and continuous experiments (part 2).

1.1 Part 1: Batch experiments

The major conclusions from the batch experiments are as follows:

NiX treatment is able to create long-term improvements in biomethane potential in a number of diverse and commercially interesting biomasses. The biomasses tested in batch assays were anaerobically digested fiber, from Morsø Bioenergi, cow deep litter, dewatered pig manure, chicken litter and hen litter. Biomethane potential improvements were 34%, 33% 27%, 22% and 2%, respectively compared to the untreated biomass.

There appears to be an inverse relationship between relative improvement and absolute methane yields, in that the biomasses producing the lowest amounts of methane without NiX treatment also showed the highest relative improvements. This indicates that the more difficult a substrate is to degrade the higher the effect of the NiX treatment.

In addition to the effect on biomethane potential, NiX treatment consistently removes 2/3 of the inorganic nitrogen, thus facilitating the use of nitrogen rich substrates for biogas production. The combination of NiX treatment with sudden pressure release and extended thermal treatment was also investigated, however the effect hereof was found to be insignificant.

1.2 Part 2: Continuous experiments

The major conclusions from the continuous pilot-scale experiment are as follows:

A CSTR biogas process with thermo-chemical treatment NiX and with chicken litter as mono-substrate was run for four months at thermophilic conditions and for five months at mesophilic conditions.

The thermophilic CSTR biogas process could run steadily when the substrate was a mixture of raw chicken litter and water at a ratio of 1:4.17 (1 kg wet weight of chicken litter and 4.17 kg of water). Applying NiX treatment to the thermophilic CSTR biogas process decreased the NH4-N (ammonium nitrogen) concentration in the digester by 15%.

Liquid re-circulation and NiX treatment were applied to the mesophilic CSTR biogas process. Extra water was added to the process only via steam condensation during NiX treatment (0.05 kg-H_2O / kg WW chicken litter). The mesophilic process did not result in stable conditions because of lack of water. As a consequence, the TS (total solids) and NH4-N concentration in the digester increased.

Addition of extra water (0.42 kg-H_2O/kg-chicken litter, including 0.05 kg H_2O / kg WW chicken litter as steam during NiX treatment) is needed to run stable biogas processes based on chicken litter as mono-substrate. The amount of water needed may change depending on the re-circulation ratio and on the organic loading rate (OLR).

During mesophilic operation, the methane yield was stable at 297 ± 35 mL CH_4 / g VS (where VS is the organic matter content of the chicken litter) and decreased when the NH4-N concentration in the digester increased beyond 6.4 g / kg WW. NiX treatment improved by 13% the methane yield of the chicken litter (measured in batches) and removed 47% of the NH4-N from the substrate.

The results obtained during the pilot-scale experiments were lower than the results obtained during Part 1 of the project (65% NH4-N removal, 22% methane yield improvement) mainly because of differences in the raw chicken litter and because of the different scale of the experiments.

Nitrogen measurements showed that the concentration of organic nitrogen does not change during NiX treatment. Approximately 30% of the organic nitrogen is converted into NH4-N during the storage of the NiX-treated mixture in the substrate tank. The biogas process resulted in 45% conversion of organic nitrogen into NH4-N.

Preliminary investigations on uric acid degradation showed that the degradation of uric acid follows the same pattern as the degradation of the overall organic nitrogen.

2 Abbreviations

AD	-	Anaerobically digested
BMP	-	Biomethane potential
CSTR	-	Continously stirred tank reactor
GEA	-	GEA westphalia mobile decanter
HRT	-	Hydraulic retention time
N	-	Nitrogen
NmL	-	Normal mL (milliliter at 1 bar and 273 K)
Org-N	-	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

3 Introduction

The major contributing factor to the power production of a biogas plant is the quality of the biomass input. Certain substrates, such as glycerol, oil and energy crops, are energy rich and/or easily degradable and are therefore desirable as substrates for biogas production. Other biomass types, such as pig and cow slurry, deep litter and poultry litter, are low in energy yield, hard to degrade and/or contain compounds inhibitory to the biogas process. Energy rich substrates are more expensive and harder to come by, and technologies that may improve on the biogas yield of the inexpensive, low energy type biomasses, are increasingly more interesting.

The major reason for the poor degradability of biomass types such as straw and manure, is their high content of lignin. Lignin is a macromolecule, which serves to uphold the rigid structure of the plant as well as offer protection against fungi, bacteria and vira. Lignin encompasses the easily degradable cellulose and hemi-cellulose in a compound called lignocellulose, and hence creates a physical barrier for the microorganisms in the anaerobic digestion process. In order to create long term improvements in biogas yield it is thus essential to attack the ligno-cellulosic structure.

The NiX technology is a registered trademark. It refers to the patented pre-treatment technology developed to attempt to overcome the problems related to lignified biomasses. NiX combines heat and alkalinity in a pressure cooking vessel to attack and dissolve the ligno-cellulosic structure as well as create an environment for removal of the inhibitory nitrogen in the process. The NiX technology comprises treatments of a biomass with saturated steam until reaching working temperatures between 100 ℃ and 220 ℃ in combination with a base.

Documentation of the technology up to now has consisted primarily of preliminary results from a full-scale pressure cooker. However, due to the need for obtaining statistically significant reproducible data, a pilot-scale pressure cooker has been developed to allow detailed investigations of the process in lab-scale batch tests as well as in pilot-scale continuous tests. In one pilot-scale test with dewatered cow manure, improvements of up to 40% were achieved when digested material from a primary reactor was pressure cooked prior to digestion in a secondary reactor.

This project has focused on investigating the effect of NiX treatment on various biomasses at varying temperatures/pressures and alkalinity. The effect of different parameters were optimised and investigated on a number of biomasses in lab-scale batch experiments, and subsequently a set of parameters were chosen to perform NiX treatment on a single biomass in pilot-scale CSTR experiment. The following sections describe the results obtained in batch and CSTR.

4 Batch investigations

4.1 Purpose

To investigate the influence of temperature/pressure, base concentration, base choice, biomass choice, aeration, and sudden pressure drop (Flash) on NiX treatment with respect to biomethane potential (BMP) enhancement and nitrogen removal.

4.2 Introduction

The parameters believed to be of greatest importance in the optimization of the NiX process are:

1. Temperature/Pressure
2. Alkaline concentration
3. Choice of base
4. Biomass type
5. Post-pressure cooking aeration and thermal treatment
6. Flash

Treatment of a biomass at elevated temperature/pressure in combination with a strong base has several potential effects. The ligno-cellulosic structure is altered and parts of it are dissolved in a manner which likely increases the amount of degradable substrate available, hence increasing the biomethane potential of the biomass. However, by doing this, there is a chance of releasing small molecules which function as inhibitors of the anaerobic digestion (AD) process. The optimal combination of temperature levels and base concentrations must be determined empirically.

Temperature and base also affects the removal of ammonium nitrogen. At higher temperatures and base concentrations the nitrogen equilibrium shifts from dissolved ammonium nitrogen ($NH_4^+{}_{(aq)}$) towards gaseous ammonium nitrogen ($NH_{3(g)}$). NH_3 is volatile and may be removed from the system via the airspace above the liquid. The manner in which pressure is released from the system also influences the removal of nitrogen. Actively releasing steam from a pressurized system causes boiling of the liquid. This in turn causes continuous replacement of the airspace volume with steam, and aids in transferring NH_3 from the liquid to the headspace. Aeration (i.e. bubbling of air through the treated sample) is another way of facilitating the removal of ammonia. Finally, sudden pressure decreases (Flash) may contribute in altering ligno-cellulosic structure resulting in higher BMP levels.

It is clear that the parameters mentioned above interlink and influence each other. However, they also exert individual effects and they may thus be addressed individually.

The batch investigations used the following experimental approach to attempt to elucidate the influence of the above mentioned parameters on BMP and nitrogen content:

4.2.1 Phase A: Pressure and base optimization

Parameters 1, 2 and 3 were addressed first. Determination of the optimal combination of temperature, base concentration and base choice on a few biomasses yielded a “standard” NiX pressure cooking method which could be used when addressing the remaining parameters.

4.2.2 Phase B: Biomass treatments and overall optimization

Parameters 4, 5 and 6 were addressed second. Determination of the robustness of the treatment using the “standard” NiX treatment on a variety of biomasses. Determination of the additive effect of Flash and extended treatment.

4.3 Materials and Methods

4.3.1 Biomass Characterization

Total solids (TS) contents were determined by heating the sample to 105 ℃ for a minimum of 24 hours. Volatile solids (VS) contents were determined by burning the sample at 550 ℃ for 3-4 hours. The substrates tested were selected based on their commercial potential (availability, cost, etc.) and composition (such that the tested substrates represented a wide variety of biomasses).

4.3.2 Nix treatment

The Nix technology consists of a thermo-chemical treatment of the substrate. Substrate is mixed with or without saturated lime ($Ca(OH)_2$) and water to the desired concentration. The treatment is performed in a pilot scale pressure cooker (see Figure 2) in which saturated steam is used to raise the pressure and temperature. After treatment pressure may be decreased either by passive cooling or by actively releasing vapor from the treatment. Samples were collected and processed according to Figure 1.

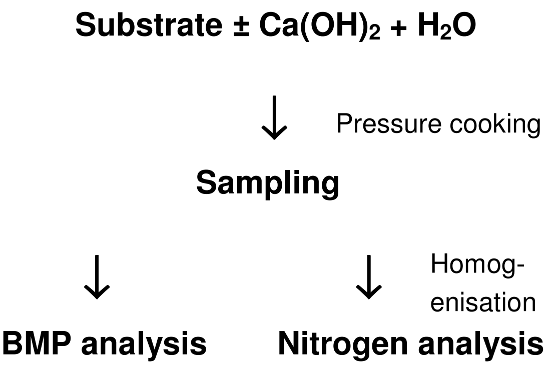

Figure 1 – Overview of treatment and analysis flow

Figure 2 - Pilot scale pressure cooker

4.3.3 Biomethane potential analysis

All BMP assays were carried out according to the German standard VDI4630 with certain alterations. Batches were prepared in 500 ml infusion glass bottles. The inoculum was taken from a thermophilic main digester at Foulum biogas plant and incubated at 52 ± 1 ℃ for 10 days before substrate addition in order to minimize the relative contribution from the inoculum to the total gas production. 200 ml of inoculum was used per bottle.

Each batch bottle was prepared by addition of approx. 1 g VS followed by addition of 200 mL inoculum (VS ~ 1%). Resulting substrate VS concentration in each of the substrate batch bottles was 5 g substrate VS/L inoculum. From each of the independent Nix treatments 3-6 replicates were incubated.

Figure 4 - Batch bottles in heat cabinet

For examination of inoculum quality, 3-6 bottles containing 0.5 g cellulose per 200 ml inoculum were incubated (positive controls). For determination of CH_4 production from the inoculum during substrate digestion, 3-6 replicate control batches of 200 ml inoculum were also incubated (blanks). After addition of inoculum and substrate all bottles were flushed with N_2, mixed and closed with gas tight rubber stoppers and aluminum screw lids before incubation at 52 ± 1 °C in heat cabinet for the duration of the batch test.

4.3.4 Measurements and Analysis

The CH_4 content in the headspace of the batch bottles was measured by GC (Perkin Elmer, Auto-analyzer XL) equipped with a capillary column (wax 0.53 mm ID, 30 m) and a FID detector (Figure 3).

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 could be 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 (mL methane per gram substrate VS added) was calculated by subtraction of background and normalizing according to VS concentration.

Figure 3 - Gas chromatograph used for CH_4 analysis

4.3.5 Nitrogen content analysis

Samples were analyzed for Total Ammonium Nitrogen (TAN) and Total Kjeldahl Nitrogen (TKN) according to the Kjeldahl method. Destruction of samples were performed on a Tecator™ Digestion Unit Auto Lift 20 and distillations were performed on a Büchi K355 distillation unit

4.4 Phase A: Pressure and base optimization

4.4.1 Experimental considerations and setup

Temperature/pressure and alkaline addition are the parameters believed to have the greatest influence on BMP and nitrogen removal. A set of experiments, with the purpose of determining the optimal temperature and base concentration, were set up. The initial experiments were performed on one biomass with varying temperatures and base concentrations (see Figure 4).

The experimental matrix consisted of 5 base concentrations, 3 temperatures/pressures and 3 replicates, making up a total of 45 different treatments to be tested. Since each pressure cooking cycle takes 3-4 hours, it was unfeasible to carry out each experimental combination individually. The experiments were thus performed without active pressure[1] release to avoid boiling of the samples, and thus allow more than one treatment per pressure cooking cycle. As a consequence, nitrogen release from this setup only occurs to a limited degree, and the chemical equilibrium will not be allowed to reach its natural levels.

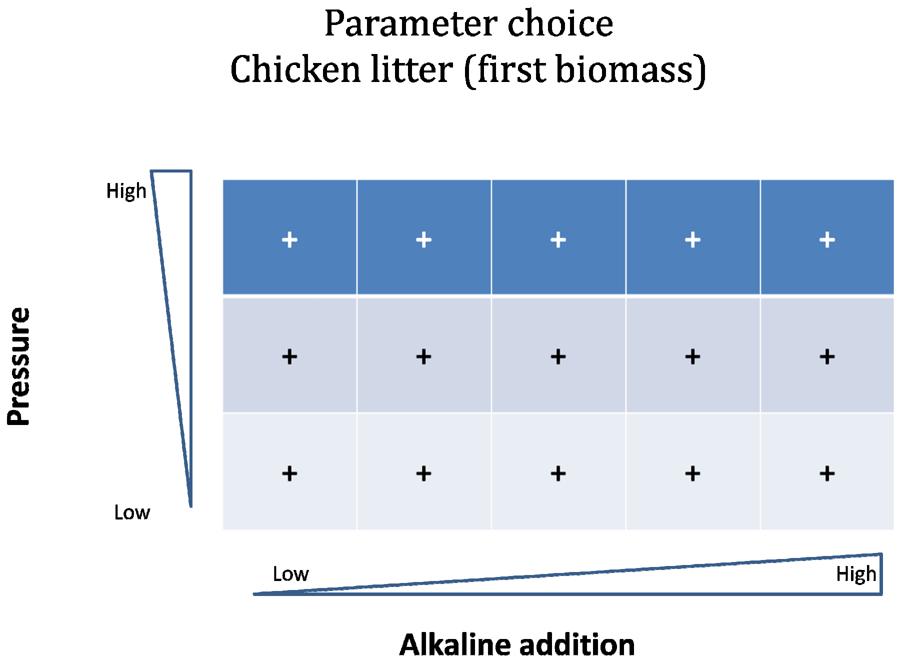

Figure 4 – Experimental matrix showing the combination of parameters tested to elucidate the optimal combination of temperature and base concentration. Pressures tested were 0 barg, 4 barg and 9 barg. Base concentrations were 0 wt%, 0,25%, 0,5 wt%, 1 wt%, and 2 wt% $Ca(OH)_2$

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[1] The pressure cooker was allowed to cool down without opening any vents to release the pressure. This passive cool down method resulted in very little boiling of the liquid.

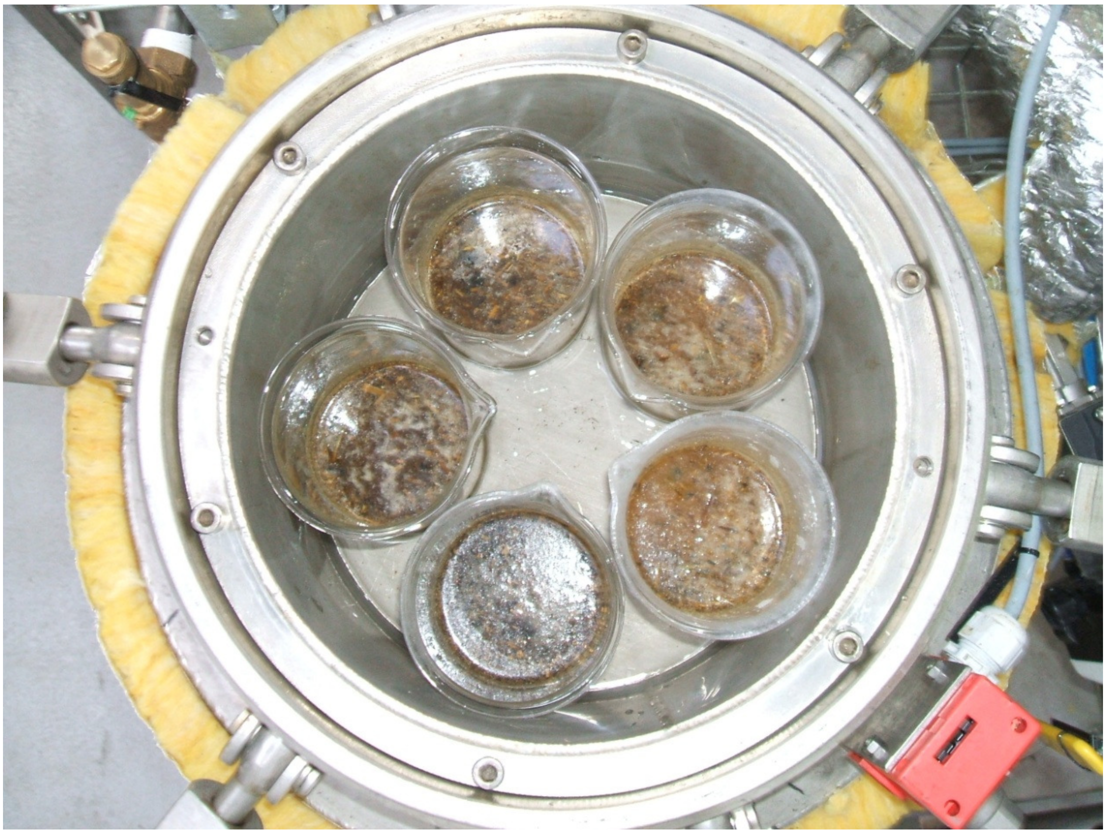

Figure 5 – View inside the pressure cooking assembly showing 500 mL beakers with chicken manure. Each beaker contains varying amounts of $Ca(OH)_2$ corresponding to either 0%, 0,25%, 0,5%, 1% or 2% (w/w).

4.4.2 Results and discussion

The results of Phase A will be briefly presented as they are relevant only for the following phases.

- No significant differences were found between two different bases (NaOH and $Ca(OH)_2$) and all following experiments were continued using the cheaper alternative $Ca(OH)_2$.
- Nitrogen removal optimum occurs at 1%-2% $Ca(OH_2)$ addition.
- BMP is slightly improved in pressure cooking of chicken litter at 4 barg without base addition.
- Base addition seems to have a negative effect on BMP.

$Ca(OH)_2$ has a low solubility, and it was therefore a possibility that the very soluble NaOH, would behave differently. However, no observable difference was found between the two compounds with respect to nitrogen removal, which may indicate that the non-dissolved $Ca(OH)_{2(s)}$ functions as a buffer and the equilibrium settles so fast that it is not a limiting factor.

It was unexpected that the addition of base to the samples seemed to lower the BMP both at 4 barg and 9 barg. This was not expected as previous preliminary result had indicated an increase in

BMP levels with addition of base. The results may however be explained with consideration to the equilibrium equation of ammonia. When ammonium turns into ammonia and leaves the system it utilizes a hydroxyl molecule. Both ammonia and hydroxyl are both potentially inhibitory to the AD process. It was considered possible that a different result might be obtainable when carrying out the experiments with active pressure release. Based on the nitrogen removal optimum a concentration of 2% was chosen for the following experiments using active pressure release.

4.5 Phase B: Biomass treatments and overall optimization

4.5.1 Experimental considerations and setup

To establish the effect of the parameter settings determined in phase A, as well as evaluate the influence of biomass type and aeration on Nix treatment a number of biomasses were treated and analyzed.

The following table shows the biomasses that were examined and the parameter settings used. More detailed descriptions of the individual biomasses may be seen under the relevant subsections.

	Pressure	**Temperature**	**Pressure hold time**	**Pressure release time**	**Sample TS**	**Sample VS**	**Extended thermal treatment/ aeration**	**Flash**
Chicken litter	4 barg	146 °C	20 min	20-30 min.	10%	8,5%	Yes/No	Yes
Cow deep litter	4 barg	146 °C	20 min	20-30 min.	10%	6,8%	Yes/No	No
GEADWP	4 barg	146 °C	20 min	20-30 min.	10%	7,7%	Yes/Yes	No
Glenrath hen litter	4 barg	146 °C	20 min	20-30 min.	10%	6,6%	No/No	No
Morsø Biogas AD fiber	4 barg	146 °C	20 min	20-30 min.	10%	7,3%	No/No	No

Table 1 – Parameter settings for the individual biomasses.

The results from NiX treatment of the different biomasses are presented below. Each section may be read individually.

4.5.2 Chicken litter

Summary and Conclusion
Chicken Litter was treated with Xergi Nix technology and analyzed for biomethane potential and nitrogen content before and after treatment. Results from untreated Chicken Litter are compared with Nix treated Chicken Litter to evaluate the effect of the treatment.

Several variations with respect to the general Nix treatment were included in the investigation of Chicken Litter:

1. Nix treatment in the absence of $Ca(OH)_2$ to elucidate the effect of saturated lime on the biological methane potential (BMP)..
2. Incubation at 70 ℃ subsequent to Nix treatment to investigate the potential effect of an extended thermal treatment on BMP[2].

BMP assays on Nix treated Chicken Litter were initiated on October 19th 2010 and were allowed to run for 48 days. However, due to technical problems only data up to day 27 are valid. BMP assays on Chicken Litter treated with Nix in the absence of saturated lime were initiated on January 17th 2011 and run for 81 days.

	Units	Untreated Chicken Litter	Nix treated Chicken Litter
BMP	NmL CH_4/g VS (NmL CH_4/g wet weight)	277 (± 19) (71)	338 (± 23) (87)
Total Ammonium Nitrogen	g N/kg VS	14,5 (± 1,3)	5,0 (± 0,2)
Total Kjeldahl Nitrogen	g N/kg VS	60,3 (± 0,9)	51,0 (± 1,3)
Total Organic Nitrogen	g N/kg VS	45,8 (± 1,6)	46,0 (± 1,3)
Total Solids content	w/w %	61,8 (± 0,3)	
Volatile solids content	w/w %	52,8 (± 0,3)	

Table 2 – Overview of key results associated with the analysis of Chicken Litter.

- NiX treatment of Chicken Litter resulted in a 22% BMP increase compared to untreated substrate.
- Over 90% of the final BMP yield in the treated sample was obtained after 7 days of incubation. The untreated sample realized over 90% of its final BMP value after 10 days of incubation.
- An extended thermal treatment at 70 ℃ for 3 days showed no effect on BMP.
- NiX treatment with Flash showed no improvement (data not shown).

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[2] Note – no aeration

- Treating Chicken Litter with Nix technology has a significant effect on BMP, and saturated lime is necessary to obtain optimal improvements.
- Nix treatment has no effect on removal of organic nitrogen, however total ammonium nitrogen levels are decreased by ~65%.
- Total nitrogen levels are reduced by 15% due to Nix treatment

Results

TS and VS analysis

TS and VS contents of the Chicken Litter were determined in triplicate prior to Nix treatment.

	Unit	Untreated Chicken Litter	
Total Solids	w/w%	61,8	± 0,3
Volatile Solids	w/w%	52,8	± 0,3

Table 3 – VS and TS levels of Chicken Litter

Figure 6 – Chicken litter

BMP analysis

Specific methane yields obtained during the BMP analysis of treated and untreated Chicken Litter are shown in Figure 7 and Figure 8. All curves show a steady increase in methane yield until day 15. Error bars represent ±1 standard deviation.

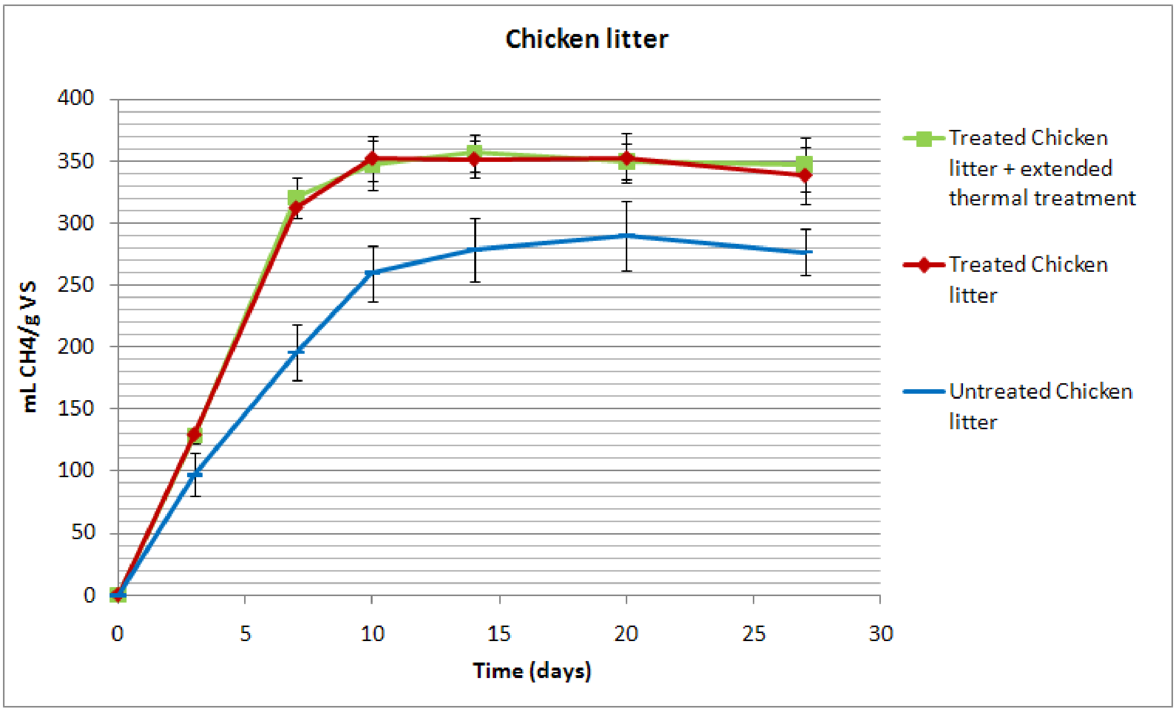

Figure 7 - Methane yield from batch bottles with untreated Chicken Litter and Nix treated Chicken Litter with or without an extended thermal treatment at 70 °C.

After 27 days BMP levels have reached 277 (± 19) NmL CH_4/g VS for the untreated Chicken Litter and 338 (± 23) NmL CH_4/g VS for treated Chicken Litter without extended thermal treatment, and 347 (± 22) NmL CH_4/g VS with extended thermal treatment (see Figure 7). There is a clear effect of Nix treatment on Chicken Litter, whereas there is no additional effect of a subsequent prolonged incubation at 70 °C.

More than 90% of the final BMP yield in the treated sample was obtained after 7 days of incubation. The untreated sample attained over 90% of its final BMP yield after 10 days of incubation. Technical problems precluded the analysis after 27 days and it is not possible to conclude on the prolonged effect of the treatment. A similar experiment showed a diminished effect of the treatment from day 20-50, however, this experiment is deemed less reliable as the absolute BMP levels are unrealistically high. It should be noted that other similar experiments show that although the improvements obtained in the Nix treatments are gradually reduced over time, they are usually still significant after 50-80 days of thermophillic digestion.

Based on a kinetic analysis of the gas production the expected yield in a full scale continuous AD plant with a 20 days thermophilic primary digestion and a 10 days mesophilic secondary digestion will be approx. 325 NmL CH4/g VS equal to 94% of the obtained batch yield.

To investigate the effect of saturated lime in Nix treatment Chicken Litter was Nix treated in the absence of $Ca(OH)_2$ (see Figure 8). This BMP assay was run for 81 days and also shows a significant effect of the treatment. The course of the graphs show a slight peak around day 15, after which the curves drop and continue on a slight increase. This type of behaviour is often observed in BMP assays with low substrate concentrations, and is believed to be caused by an initial over-production of methane from the inoculum VS, which evens out over time as the inoculum controls catch up. The highest improvements are obtained between 10 and 20 days, after which the effect is very gradually reduced.

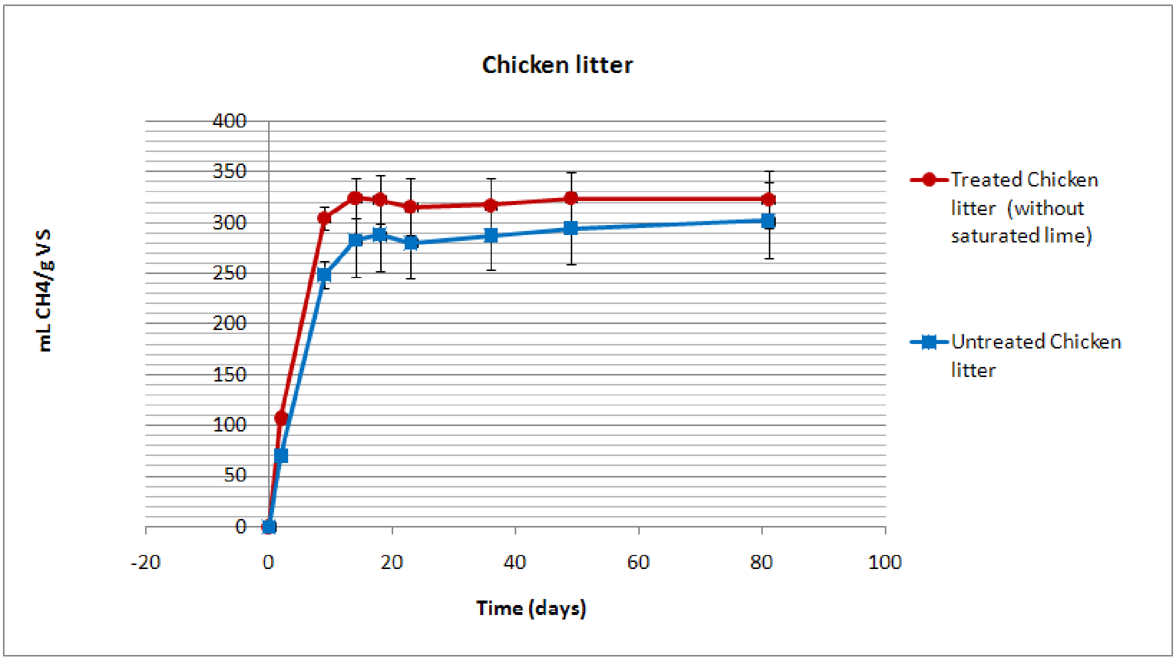

Figure 8 - Methane yield from batch bottles with untreated Chicken Litter and Nix treated Chicken Litter in the absence of saturated lime. [3]

Nitrogen analysis

The nitrogen content of treated and untreated Chicken Litter are shown in Figure 9. Specific values and the resulting nitrogen reductions as a result of Nix treatment are shown in Table 4.
Inorganic nitrogen constitutes about a quarter of the entire nitrogen pool (~24%). Organic nitrogen constitutes the remaining ~76%.

Using Nix technology inorganic nitrogen is removed to an absolute level of 5,0 g N/kg VS, which constitutes a 65% reduction in TAN. Organic nitrogen is not removed in this treatment. Subsequent prolonged thermal treatment does not remove further nitrogen.

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[3] Methane yields have been normalized according to an internal standard

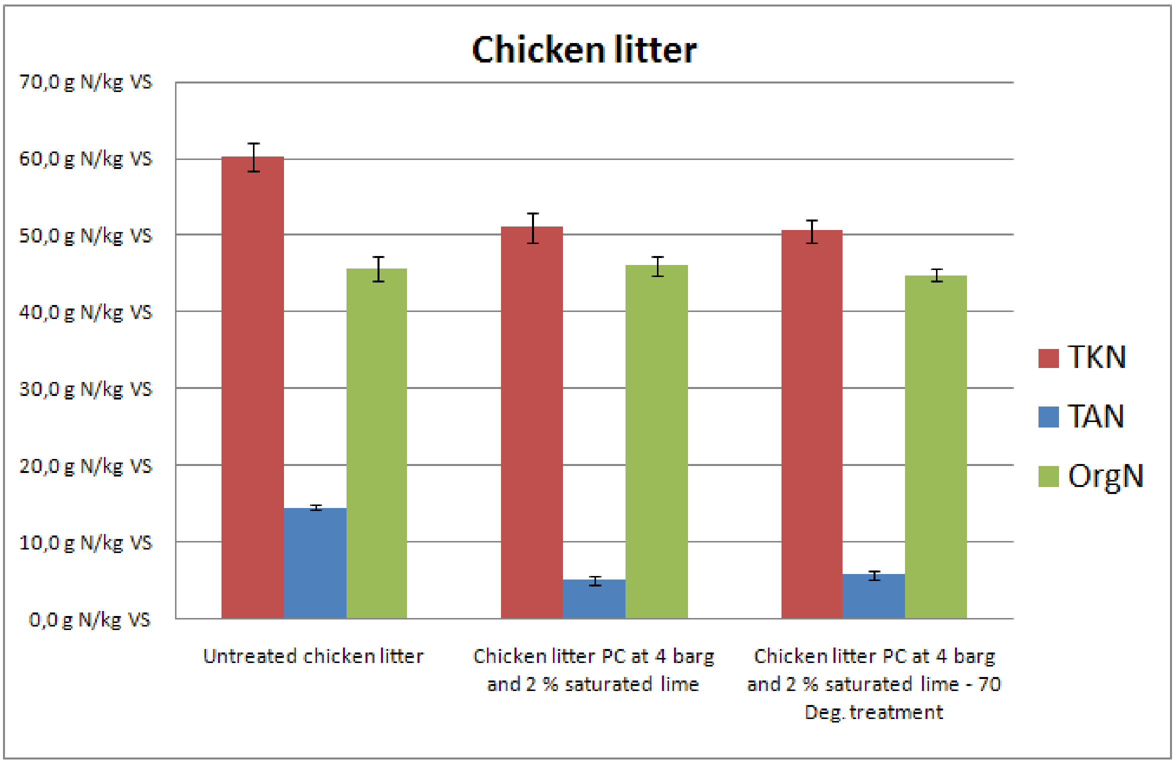

Figure 9 – Graphical representation of TKN, TAN and Organic nitrogen (Org-N) levels in untreated and treated Chicken Litter. Org-N is calculated from TKN and TAN levels (Org-N = TKN – TAN). PC = Pressure Cooked.

Total ammonium nitrogen content	TAN (g N/kg VS)	SD (g N/kg VS)	RSD %	Reduction (g N/kg VS)	Reduction in percent
Untreated Chicken Litter	14,5	1,3	9%		
Treated Chicken Litter	5,0	0,2	5%	9,5	65%
+ 70 °C for 3 days	5,7	0,4	7%	8,8	61%

Total Kjeldahl nitrogen content	TKN (g N/kg VS)	SD (g N/kg VS)	RSD%	Reduction (g N/kg VS)	Reduction in percent
Untreated Chicken Litter	60,3	0,9	1%	0,0	
Treated Chicken Litter	51,0	1,3	3%	9,2	15%
+ 70 °C for 3 days	50,6	0,6	1%	9,7	16%

Total organic nitrogen content	Org-N (g N/kg VS)	SD (g N/kg VS)	RSD%	Reduction (g N/kg VS)	Reduction in percent
Untreated Chicken Litter	45,8	1,6	3%		
Treated Chicken Litter	46,0	1,3	3%	-0,3	-1%
+ 70 °C for 3 days	44,9	0,7	2%	0,9	2%

Table 4 – Values of TAN, TKN and Org-N with standard deviations (SD), relative standard deviations (RSD) and reductions as a consequence of Nix treatment. Negative reduction values reflect a value that is higher than the untreated organic nitrogen pool. Note that the extended thermal treatment is without aeration.

4.5.3 Cow deep litter

Summary and Conclusion

Cow Deep Litter was treated with Xergi Nix technology and analyzed for biomethane potential and nitrogen content before and after treatment. Results from untreated Cow Deep Litter is compared with Nix treated Cow Deep Litter to evaluate the effect of the treatment.

Several variations with respect to the general Nix treatment were included in the investigation of Cow Deep Litter:

1. Nix treatment in the absence of $Ca(OH)_2$ to elucidate the effect of saturated lime on the biological methane potential (BMP).
2. Incubation at 70 °C subsequent to Nix treatment to investigate the potential effect of an extended thermal treatment on BMP[4].

BMP assays on Nix treated Cow Deep Litter were initiated on October 19th 2010 and were allowed to run for 48 days. However, due to technical problems only data up to day 27 are valid. BMP assays on Cow Deep Litter treated with Nix in the absence of saturated lime were initiated on January 17th 2011 and run for 80 days.

	Units	Untreated Cow Deep Litter	Nix treated Cow Deep Litter
BMP	NmL CH_4/g VS (NmL CH_4/g wet weight)	252 (± 24) (63)	334 (± 18) (84)
Total Ammonium Nitrogen	g N/kg VS	3,6 (± 0,1)	1,3 (± 0,1)
Total Kjeldahl Nitrogen	g N/kg VS	18,8 (± 0,6)	18,2 (± 0,3)
Total Organic Nitrogen	g N/kg VS	15,2 (± 0,6)	16,9 (± 0,3)
Total Solids content	w/w %	37,1 (± 1,1)	
Volatile solids content	w/w %	25,0 (± 3,1)	

Table 5 – Overview of key results associated with the analysis of Cow Deep Litter.

- NiX treatment of Cow deep litter resulted in a 33% BMP increase compared to untreated substrate.
- Close to final BMP yields were obtained in the treated sample after 10 days of incubation. The untreated sample realized most of its final BMP yield after 20 days of incubation.
- An extended thermal treatment at 70 °C for 3 days showed no effect on BMP.

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[4] Note – no aeration

- Treating Cow Deep Litter with Nix technology has a significant effect on BMP. Due to technical problems analysis was not possible after 27 days, and determination of the time span of the beneficial effects of the treatment are not possible at this time. However, other similar experiments have shown lasting effects up to 90 days.
- Nix treatment has no effect on removal of organic nitrogen, however total ammonium nitrogen levels are decreased by ~64%, corresponding to a reduction in total nitrogen of 12%. Measured TKN levels before and after show a reduction in TKN of 3%. However, Cow Deep Litter is a very inhomogeneous substrate, from which it is much easier to measure TAN than TKN, and it is thus likely that the measured TKN level after treatment is a result of the heterogenic nature of the substrate. This is corroborated by TKN results on Nix treated Cow Deep Litter, showing a total TKN reduction of 15%.

Results

TS and VS analysis

TS and VS contents of the Cow Deep Litter were determined in triplicate prior to Nix treatment.

	Unit	Untreated Cow Deep Litter	
Total Solids	w/w%	37,1	± 1,1
Volatile Solids	w/w%	25,0	± 3,1

Table 6 – VS and TS levels of Cow Deep Litter

Figure 10 - Cow Deep Litter

BMP analysis

Specific methane yields obtained during the BMP analysis of treated and untreated Cow Litter are shown in Figure 11 - Methane yield from batch bottles with untreated Cow Deep Litter and Nix treated Cow Deep Litter with or without an extended thermal treatment at 70 °C.

and Figure 12. All curves show a steady increase in methane yield until day 15. Error bars represent ±1 standard deviation.

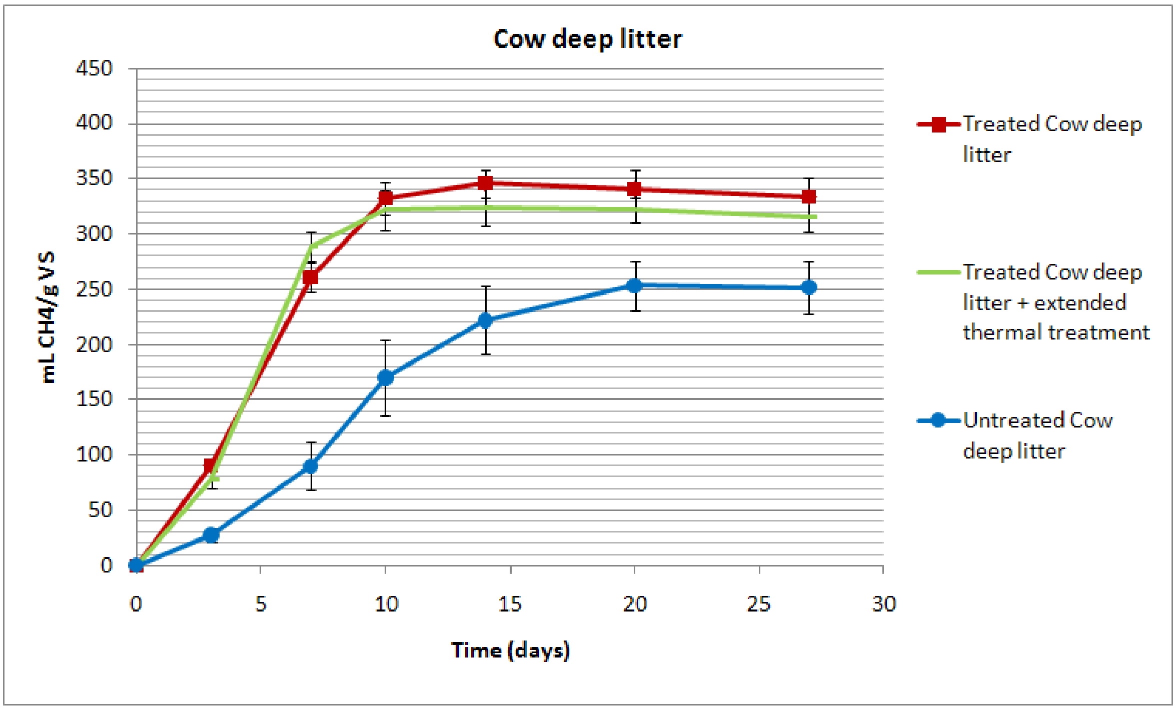

Figure 11 - Methane yield from batch bottles with untreated Cow Deep Litter and Nix treated Cow Deep Litter with or without an extended thermal treatment at 70 °C[5].

After 27 days BMP levels have reached 252 (± 24) NmL CH_4/g VS for the untreated Cow Deep Litter and 334 (± 18) NmL CH_4/g VS for treated Cow Deep Litter without extended thermal treatment, and 315 (± 13) NmL CH_4/g VS with extended thermal treatment (see Figure 11 - Methane yield from batch bottles with untreated Cow Deep Litter and Nix treated Cow Deep Litter with or without an extended thermal treatment at 70 °C.

). There is a clear effect of Nix treatment on Cow Deep Litter, whereas there is no significant additional effect of a subsequent prolonged incubation at 70 °C.

In the treated sample the final BMP yield was obtained after approximately 10 days incubation. The untreated sample required 20 days of incubation to attain its final BMP. Technical problems precluded the analysis after 27 days and it is not possible to conclude on the prolonged effect of the treatment. It should be noted that other similar experiments show that, although the improvements obtained in the Nix treatments are gradually reduced over time, they are usually still significant after 30-80 days of thermophillic digestion.

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[5] Methane yields have been normalized according to an internal standard

Based on a kinetic analysis of the gas production the expected yield in a full scale continuous AD plant with a 20 days thermophilic primary digestion and a 10 days mesophilic secondary digestion will be approx. 317 NmL CH4/g VS equal to 92% of the obtained batch yield.

To investigate the effect of saturated lime in Nix treatment Cow Deep Litter was Nix treated in the absence of $Ca(OH)_2$ (see Figure 12). This BMP assay was run for 80 days and also shows a significant effect of the treatment. The highest improvements are obtained between 15 and 20 days, after which the effect is gradually reduced. After 80 days there is no longer any significant effect of the treatment.

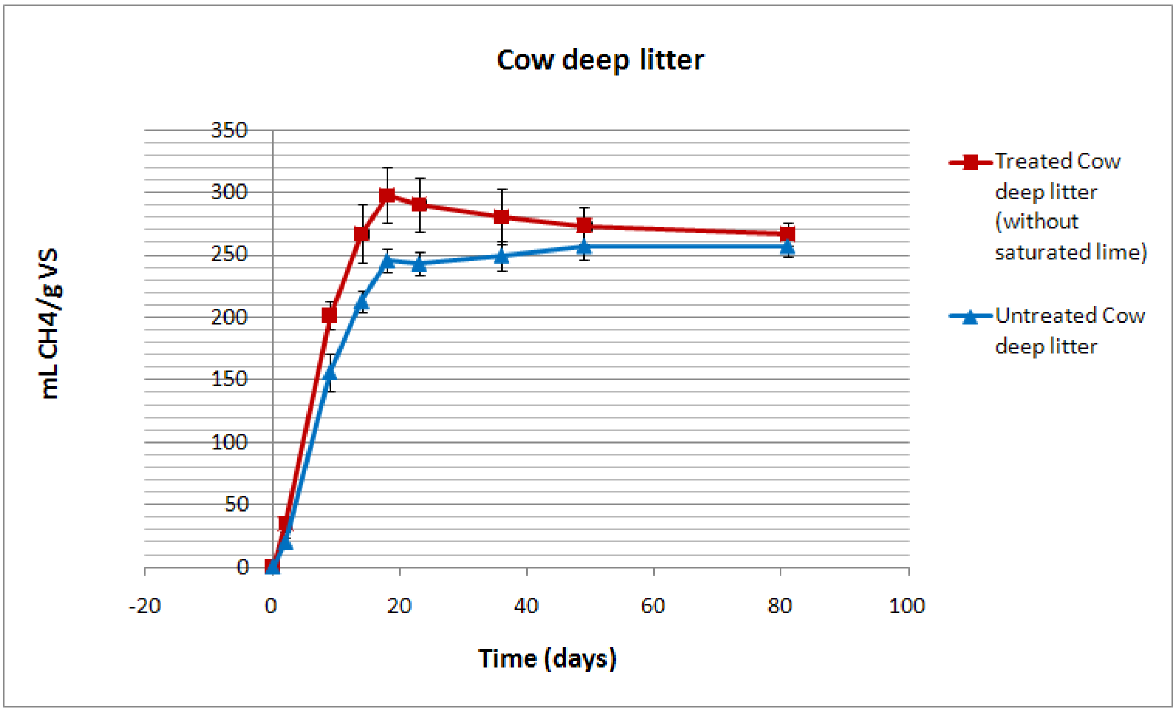

Figure 12 - Methane yield from batch bottles with untreated Cow Deep Litter and Nix treated Cow Deep Litter in the absence of saturated lime. [6]

Nitrogen analysis

The nitrogen content of treated and untreated Cow Deep Litter are shown in **Fejl! Henvisningskilde ikke fundet.**. Specific values and the resulting nitrogen reductions as a result of Nix treatment are shown in Table 7.

Inorganic nitrogen constitutes about one fifth of the entire nitrogen pool (~19%). Organic nitrogen constitutes the remaining ~81%.

[6] Methane yields have been normalized according to an internal standard

Using Nix technology inorganic nitrogen is removed to an absolute level of 3,6 g N/kg VS, which constitutes a 64% reduction in TAN. Such a reduction in TAN alone should lead to a TKN reduction of 12%. However, measured TKN levels before and after only show a reduction in TKN of 3%. Cow Deep Litter is a very inhomogeneous substrate, from which it is much easier to measure TAN than TKN, and it is likely that the measured TKN level after treatment is a result of the heterogeneous nature of the substrate. This is corroborated by TKN results on Nix treated Cow Deep Litter, showing a total TKN reduction of 15% (see **Fejl! Henvisningskilde ikke fundet.**). From this we may conclude that organic nitrogen is not removed in this treatment.

When exposing Cow Deep Litter to a prolonged thermal treatment subsequent to Nix treatment there seems to be a small effect on TAN removal. This is in contrast to previous results on GEA dewatered pig manure and Chicken litter, which show that a post Nix thermal treatment at 70 °C has no effect on TAN removal.

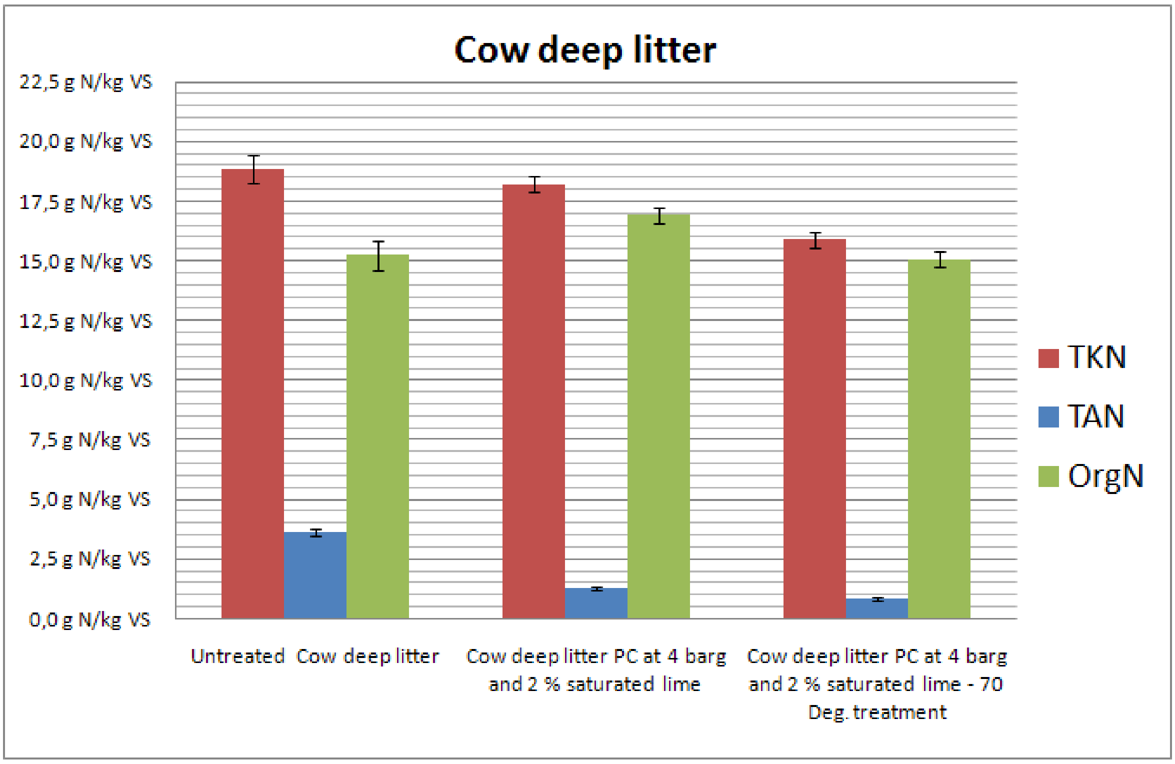

Figure 13 – Graphical representation of TKN, TAN and Organic nitrogen (Org-N) levels in untreated and treated Cow Deep Litter. Org-N is calculated from TKN and TAN levels (Org-N = TKN – TAN). PC = Pressure Cooked.

Total ammonium nitrogen content	**TAN (g N/kg VS)**	**SD (g N/kg VS)**	**RSD %**	**Reduction (g N/kg VS)**	**Reduction in percent**
Untreated Cow Deep Litter	3,6	0,1	4%		
Treated Cow Deep Litter	1,3	0,1	6%	2,3	64%
+ 70 °C for 3 days	0,8	0,0	6%	2,8	77%

Total Kjeldahl nitrogen content	**TKN (g N/kg VS)**	**SD (g N/kg VS)**	**RSD%**	**Reduction (g N/kg VS)**	**Reduction in percent**
Untreated Cow Deep Litter	18,8	0,6	3%	0,0	
Treated Cow Deep Litter	18,2	0,3	2%	0,6	3%
+ 70 °C for 3 days	15,9	0,3	2%	2,9	16%

Total organic nitrogen content	**Org-N (g N/kg VS)**	**SD (g N/kg VS)**	**RSD%**	**Reduction (g N/kg VS)**	**Reduction in percent**
Untreated Cow Deep Litter	15,2	0,6	4%		
Treated Cow Deep Litter	16,9	0,3	2%	-1,7	-11%
+ 70 °C for 3 days	15,1	0,3	2%	0,2	1%

Table 7 – Values of TAN, TKN and Org-N with standard deviations (SD), relative standard deviations (RSD) and reductions as a consequence of Nix treatment. Negative reduction values reflect a value that is higher than the untreated organic nitrogen pool. Note that the extended thermal treatment is without aeration.

4.5.4 GEA Dewatered Pig Manure

Summary and Conclusion

GEA Dewatered Pig Manure is the fiber fraction after dewatering using a GEA decanter and will be referred to as “pig fibers” in the following. The pig fibers were treated with Xergi Nix technology and analyzed for biomethane potential and nitrogen content before and after treatment. Results from untreated pig fibers are compared with Nix treated pig fibers to evaluate the effect of the treatment.

Several variations with respect to the general Nix treatment were included in the investigation of pig fibers:

1. Nix treatment in the absence of $Ca(OH)_2$to elucidate the effect of saturated lime on the biological methane potential (BMP)..
2. Incubation at 70 °C subsequent to Nix treatment to investigate the potential effect of an extended thermal treatment on BMP.
3. Incubation at 70 °C and aeration subsequent to Nix treatment to investigate the potential effect on removal of nitrogen.

BMP assays on Nix treated pig fibers were initiated on October 19th 2011 and were allowed to run for 48 days. However, due to technical problems only data up to day 27 are valid. BMP assays on pig fibers treated with Nix in the absence of saturated lime were initiated on January 17th 2011 and run for 81 days.

	Units	Untreated pig manure	Nix treated pig fibers
BMP	NmL CH_4/g VS (NmL CH_4/g wet weight)	260 (± 19) (67)	331 (± 17)[7] (85)
Total Ammonium Nitrogen	g N/kg VS	18,4 g (± 0,3)	6,7 g (± 0,5)[8]
Total Kjeldahl Nitrogen	g N/kg VS	39,9 (± 1,8)	27,9 (± 1,9)
Total Organic Nitrogen	g N/kg VS	21,5 (± 1,8)	21,2 (± 1,9)
Total Solids content	w/w %	33,4 (± 0,1)	
Volatile solids content	w/w %	25,6 (± 0,1)	

Table 8 – Overview of key results associated with the analysis of pig fibers

- NiX treatment of pig fibers resulted in a 27% BMP increase compared to untreated substrate.
- Final BMP levels are reached after 20 days of incubation in both treated and untreated samples.

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[7] After 27 days

[8] With aeration and extended thermal treatment for 3 days TAN is reduced to 4,0 (±1,4) g N/kg VS

- An extended thermal treatment at 70 °C for 3 days showed no effect on BMP.
- Treating pig fibers with Nix technology has a significant effect on BMP, and saturated lime seems to be necessary to obtain optimal improvements. The beneficial effect of Nix treatment is gradually diminished over time in the samples treated without saturated lime. Whether this is also the case for the samples containing saturated lime, is not possible to conclude due to technical problems. However, after 27 days the improvement on BMP shows no tendency to fade.
- Nix treatment has no effect on removal of organic nitrogen, however total ammonium nitrogen levels are decreased by ~64%.
- Total nitrogen levels are reduced by 30% due to Nix treatment

Figure 14 - GEA dewatered Pig Manure

Results

TS and VS analysis

TS and VS contents of the pig fibers were determined in triplicate prior to Nix treatment.

	Unit	Untreated Pig Manure	
Total Solids	w/w%	33,4	± 0,1
Volatile Solids	w/w%	25,6	± 0,1

Table 9 – VS and TS levels of GEA dewatered Pig Manure

BMP analysis

Specific methane yields obtained during the BMP analysis of treated and untreated pig fibers Pig Manure are shown in Figure 15 and Figure 16. All curves show a steady increase in methane yield until day 15. Error bars represent ±1 standard deviation.

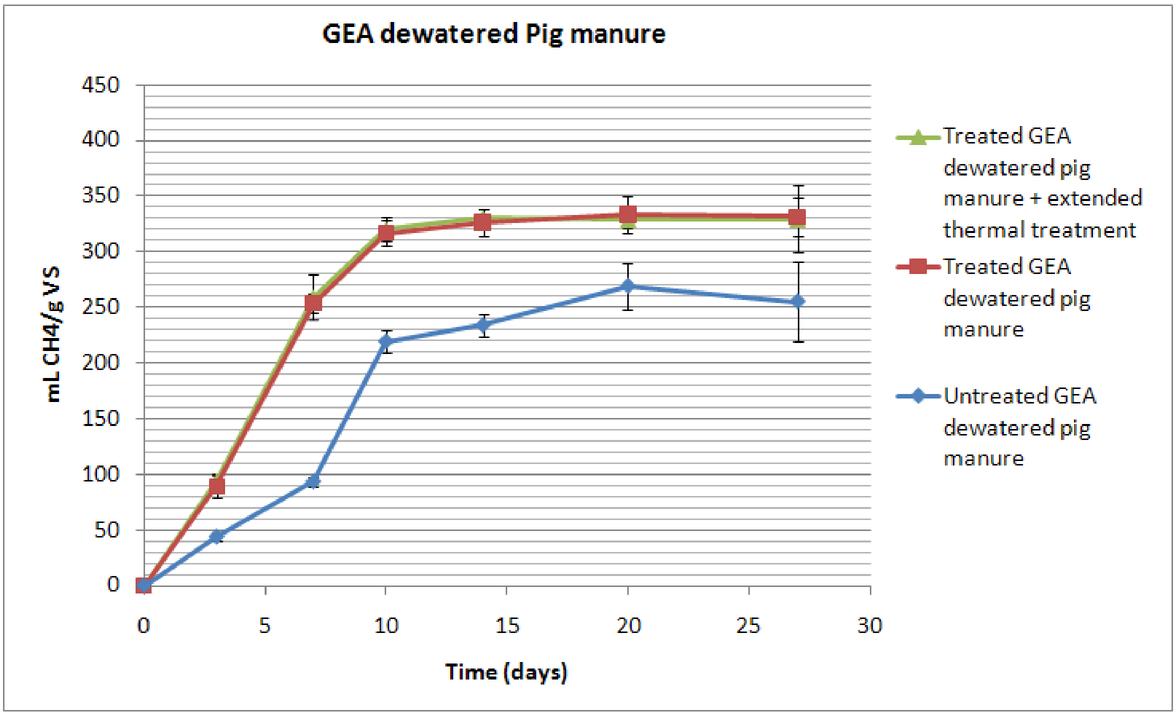

Figure 15 - Methane yield from batch bottles with untreated and NiX treated pig fibers with or without an extended thermal treatment at 70 °C[9].

After 27 days BMP levels have reached 260 (± 19) NmL CH_4/g VS for the untreated pig manure and 331 (± 17) NmL CH_4/g VS for treated pig manure without extended thermal treatment, and 329 (± 30) NmL CH_4/g VS with extended thermal treatment (se Figure 15). There is a clear effect of Nix treatment on pig fibers, whereas there is no additional effect of a subsequent prolonged incubation at 70 °C.

Final BMP yields are reached before day 20 in both treated and untreated samples. Technical problems precluded the analysis after 27 days and it is not possible to conclude on the prolonged effect of the treatment. However, other similar experiments show that, although the improvements obtained in the Nix treatments are gradually reduced over time, they are usually still significant after 50-80 days of thermophillic digestion.

Based on a kinetic analysis of the gas production the expected yield in a full scale continuous AD plant with a 20 days thermophilic primary digestion and a 10 days mesophilic secondary digestion will be approx. 305 NmL CH4/g VS equal to 91% of the obtained batch yield.

To investigate the effect of saturated lime in Nix treatment pig fibers were Nix treated in the absence of $Ca(OH)_2$ (see Figure 16). This BMP assay was run for 81 days and shows a significant

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[9] Methane yields have been normalized according to an internal standard

effect of the treatment. The course of the graphs show a peak around day 15, after which the curves drop and stabilise at the current level. This type of behaviour is often observed in BMP assays with low substrate concentrations, and is believed to be caused by an initial overproduction of methane from the inoculum VS, which evens out over time as the inoculum controls catch up.

The highest improvements are obtained between 15 and 20 days, after which the effect is gradually reduced. After 36-49 days BMP levels have reached 259 (± 23) NmL CH_4/g VS for the untreated pig manure and 288 (± 42) NmL CH_4/g VS for treated pig manure without added $Ca(OH)_2$ (see Figure 16). After 81 days there is no effect of the treatment.

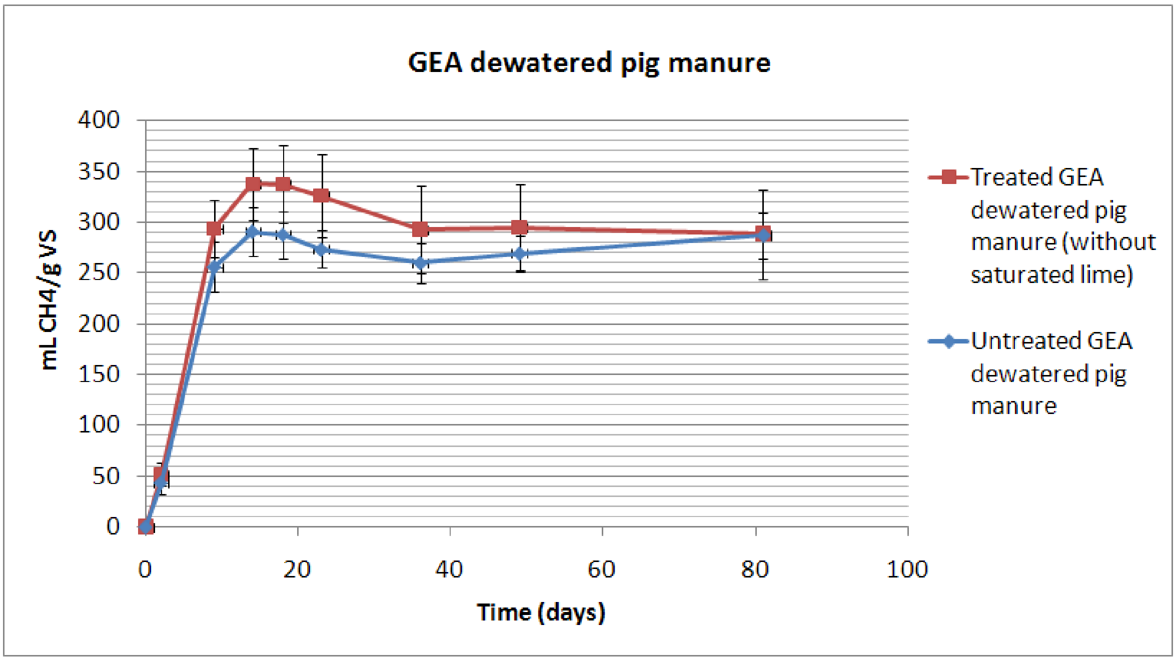

Figure 16 - Methane yield from batch bottles with untreated pig Manure and Nix treated pig manure in the absence of saturated lime.[10]

Nitrogen analysis

The nitrogen content of treated and untreated pig fibers are shown in Figure 17. Specific values and the resulting nitrogen reductions as a result of Nix treatment are shown Table 10.

Inorganic nitrogen constitutes a little less than half of the entire nitrogen pool (~46%). Organic nitrogen constitutes the remaining ~54%. Using Nix technology inorganic nitrogen is removed to an absolute level of 6,7 g N/kg VS, which constitutes a 64% reduction in TAN. Organic nitrogen is not removed in this treatment. When exposing the Nix treated pig manure to aeration at elevated temperatures TAN is further reduced to an absolute level of 4 g N/kg VS corresponding to a 78% reduction. However, high standard deviations preclude a formal conclusion on this result.

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[10] Methane yields have been normalized according to an internal standard

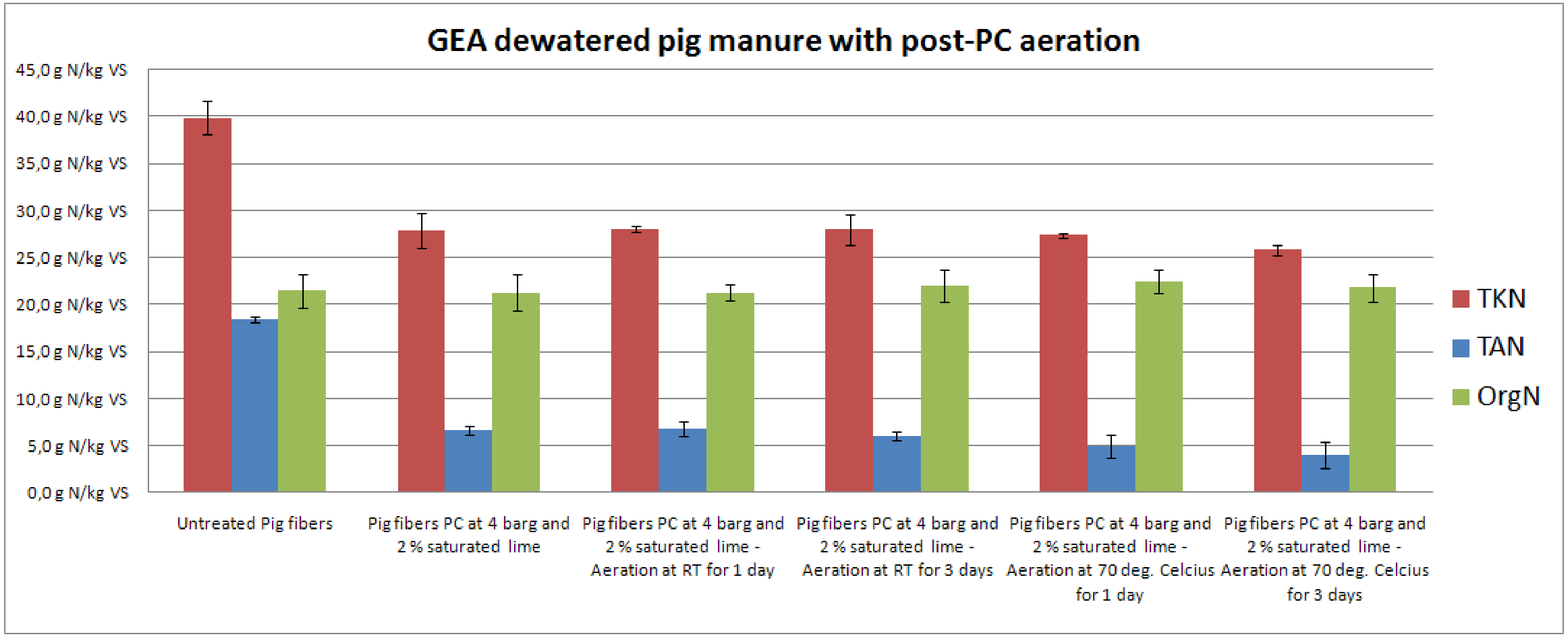

Figure 17 – Graphical representation of TKN, TAN and Organic nitrogen (Org-N) levels in untreated and treated pig fibers. Org-N is calculated from TKN and TAN levels (Org-N = TKN – TAN). PC = Pressure Cooked.

Total ammonium nitrogen content	TAN (g N/kg VS)	SD (g N/kg VS)	RSD %	Reduction (g N/kg VS)	Reduction in percent
Untreated GEA dewatered Pig Manure	18,4	0,3	2%		
Treated pig fibers	6,7	0,5	8%	11,7	64%
+ Aeration at RT for 1 day	6,8	0,8	11%	11,6	63%
+ Aeration at RT for 3 days	6,1	0,5	8%	12,3	67%
+ Aeration at 70 °C for 1 day	4,9	1,2	24%	13,5	73%
+ Aeration at 70 °C for 3 days	4,0	1,4	34%	14,4	78%

Total kjeldahl nitrogen content	TKN (g N/kg VS)	SD (g N/kg VS)	RSD%	Reduction (g N/kg VS)	Reduction in percent
Untreated GEA dewatered Pig Manure	39,9	1,8	4%		
Treated pig fibers	27,9	1,9	7%	12,0	30%
+ Aeration at RT for 1 day	28,1	0,3	1%	11,8	30%
+ Aeration at RT for 3 days	28,0	1,6	6%	11,9	30%
+ Aeration at 70 °C for 1 day	27,3	0,3	1%	12,5	31%
+ Aeration at 70 °C for 3 days	25,8	0,5	2%	14,1	35%

Total organic nitrogen content	Org-N (g N/kg VS)	SD (g N/kg VS)	RSD%	Reduction (g N/kg VS)	Reduction in percent
Untreated GEA dewatered Pig Manure	21,5	1,8	8%		
Treated pig fibers	21,2	1,9	9%	0,2	1%
+ Aeration at RT for 1 day	21,3	0,8	4%	0,2	1%
+ Aeration at RT for 3 days	21,9	1,7	8%	-0,5	-2%
+ Aeration at 70 °C for 1 day	22,4	1,2	5%	-0,9	-4%
+ Aeration at 70 °C for 3 days	21,8	1,5	7%	-0,3	-1%

Table 10 – Values of TAN, TKN and Org-N with standard deviations (SD), relative standard deviations (RSD) and reductions as a consequence of Nix treatment. Negative reduction values reflect a value that is higher than the untreated organic nitrogen pool.

4.5.5 Morsø Biogas AD Fibers

Summary and Conclusion

Anaerobically digested (AD) fibers from Morsø biogas plant was treated with Xergi Nix technology and analyzed for biomethane potential and nitrogen content before and after treatment. Results from untreated AD fibers are compared with Nix treated AD fibers to evaluate the effect of the treatment.

BMP assays were initiated December 10th 2010 and run for 88 days.

	Units	Untreated AD fibers	Treated AD fibers
BMP	NmL CH_4/g VS (NmL CH_4/g wet weight)	231 (± 18) (45)	310 (± 43) (60)
Total Ammonium Nitrogen	N/kg VS	35,9 g (± 0,2)	13,3 g (± 3,2)
Total Kjeldahl Nitrogen	N/kg VS	48,8 (± 0,2)	29,2 (± 3,2)
Total Organic Nitrogen	N/kg VS	12,9 (± 0,3)	15,9 (± 4,5)
Total Solids content	w/w %	27,8 (± 1,37)	
Volatile solids content	w/w %	19,5 (± 0,5)	

Table 11 – Overview of key results associated with the analysis of AD fibers.

- NiX treatment AD fibers resulted in a 34% BMP increase compared to untreated substrate.
- Approximately 90% of the final BMP yield in the treated sample was obtained after 33 days of incubation. The untreated sample had only reached 86% of its final BMP value after 53 days of incubation.
- Treating the AD fibers with Nix technology has a significant and lasting effect on BMP
- Nix treatment has no effect on removal of organic nitrogen, however ammonium/ammonia levels are decreased by >60%.
- Total nitrogen levels are reduced by 40% due to Nix treatment.

Results

TS and VS analysis

TS and VS contents of the AD fibers were determined in triplicate prior to Nix treatment.

	Unit	Untreated AD fiber	
Total Solids	w/w%	27,8	± 1,37
Volatile Solids	w/w%	19,5	± 0,50

Table 12 – VS and TS levels of AD fibers.

Figure 18 – Morsø Bioenergi AD fiber

BMP analysis

Specific methane yields obtained during the BMP analysis of treated and untreated AD fibers are shown in Figure 19. All curves show a steady increase in gas yield until day 88. Error bars represent ±1 standard deviation.

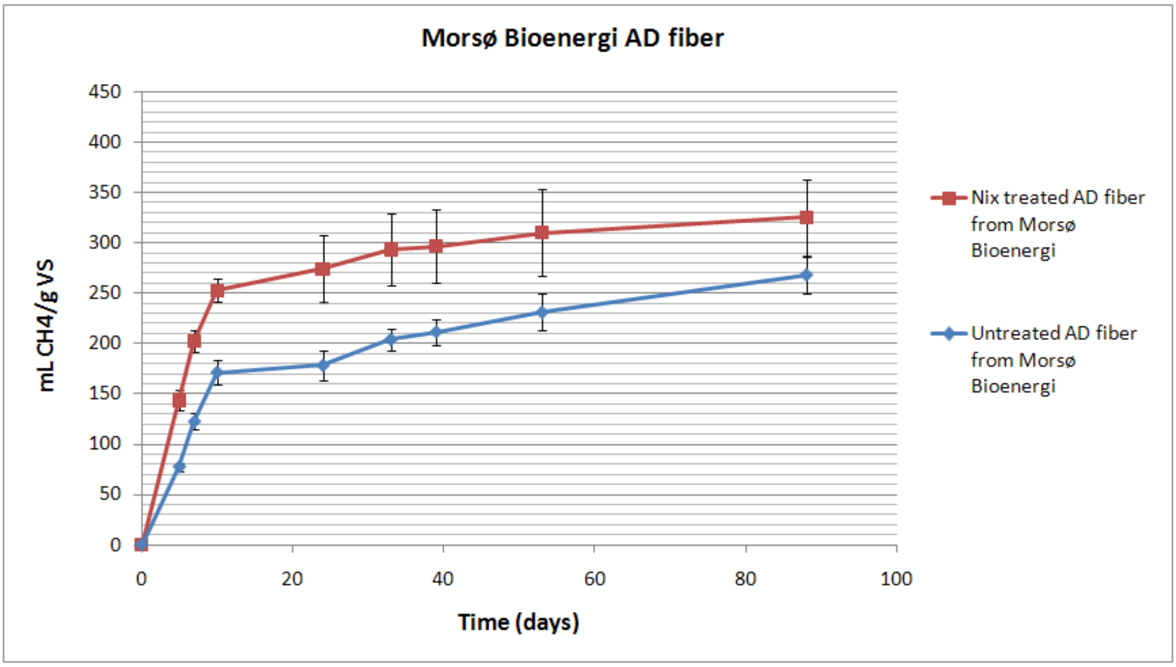

Figure 19 - Methane yield from batch bottles with treated and untreated AD fiber.

After 88 days BMP levels have reached 268 (± 18) NmL CH_4/g VS for the untreated AD fiber and 325 (± 38) NmL CH_4/g VS for treated AD fiber. On day 33 app. 90% of the final yield had been obtained in the treated sample. The same relative level was obtained after 53 days in the untreated sample. The effect of the Nix treatment on BMP seems to decrease after prolonged incubation.

However since HRT values typically are lower than 88 days, this is unlikely to have any significant practical consequence. Both treated and untreated samples continue producing gas even after 88 days, which is in contrast to most substrates which reach their maximal BMP value after 4-6 weeks incubation. However, this may be explained by the fact that the Morsø Bioenergi AD fiber has been both anaerobically digested and dewatered prior to Nix treatment and analysis. The content of easily digestible organic compounds is thus likely to be very low, with longer digestion times as a result.

Based on a kinetic analysis of the gas production the expected yield in a full scale continuous AD plant with a 20 days thermophilic primary digestion and a 10 days mesophilic secondary digestion will be approx. 245 NmL CH4/g VS equal to 78% of the obtained batch yield.

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Nitrogen analysis

The nitrogen content of treated and untreated AD fibers are shown in **Fejl! Henvisningskilde ikke fundet.**. Specific values and the resulting nitrogen reductions as a result of Nix treatment are shown in Table 13.

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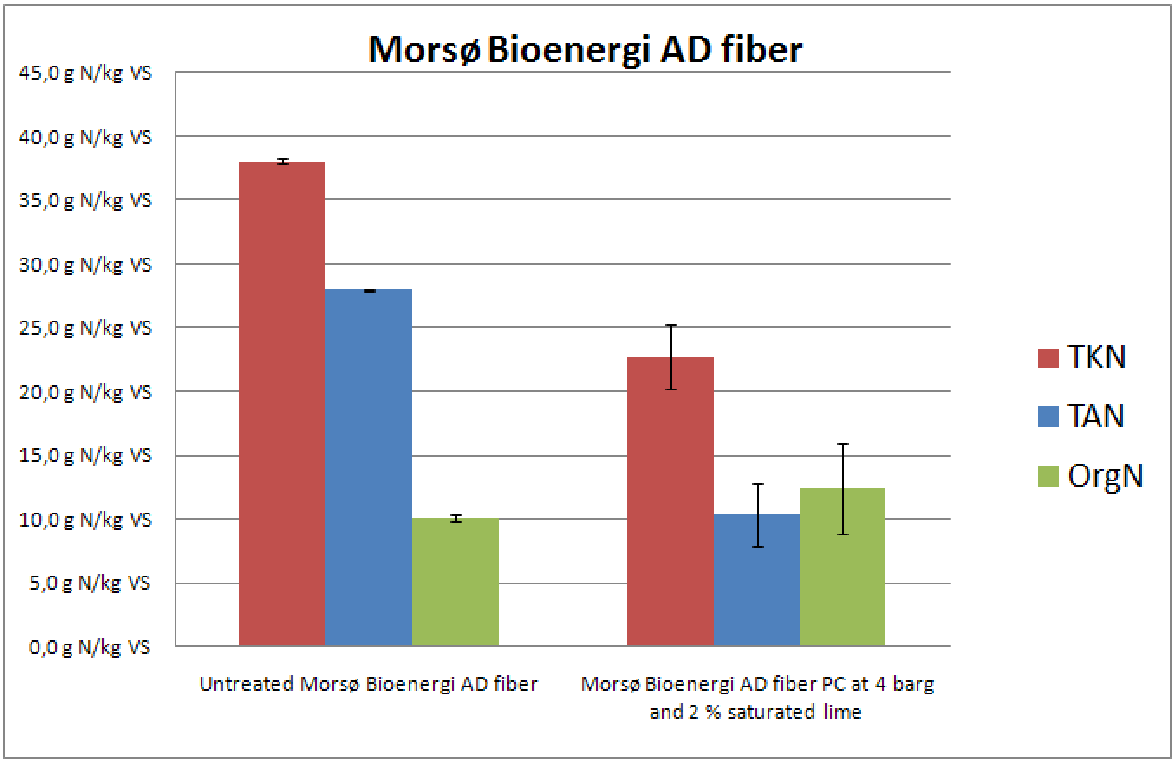


Figure 20 – Graphical representation of TKN, TAN and Organic nitrogen (Org-N) levels in untreated and treated AD fiber. Org-N is calculated from TKN and TAN levels (Org-N = TKN – TAN). PC = Pressure Cooked.

Total ammonium nitrogen content	TAN (g N/kg VS)	SD (g N/kg VS)	RSD %	Reduction (g N/kg VS)	Reduction in percent
Untreated AD fibers	35,9	0,2	0,4%		
Treated AD fibers	13,3	3,2	24%	22,6	63%

Total kjeldahl nitrogen content	TKN (g N/kg VS)	SD (g N/kg VS)	RSD %	Reduction (g N/kg VS)	Reduction in percent
Untreated AD fibers	48,8	0,2	0%		
Treated AD fibers	29,2	3,2	11%	19,7	40%

Total organic nitrogen content	Org-N (g N/kg VS)	SD (g N/kg VS)	RSD %	Reduction (g N/kg VS)	Reduction in percent
Untreated AD fibers	12,9	0,3	2%		
Treated AD fibers	15,9	4,5	28%	-2,9	-23%

Table 13 – Values of TAN, TKN and Org-N with standard deviations (SD), relative standard deviations (RSD) and reductions as a consequence of Nix treatment. Negative reduction values reflect a value that is higher than the untreated organic nitrogen pool.

Inorganic nitrogen constitutes a significant amount of the entire nitrogen pool (~73%). Organic nitrogen constitutes the remaining ~27%. With Nix technology inorganic nitrogen is removed to an absolute level of 13 g N/kg VS, which constitutes a 63% reduction. Since Nix technology is able to reduce TAN levels to 4-6 g N/kg VS in most substrates (with TAN levels of up to ~20 g N/kg VS in untreated substrate), it is likely that an optimization of the procedure will lead to even higher removal levels. This is corroborated by the pH after treatment (data not shown) which indicates that most of the added base is utilised in the removal of ammonium.

4.5.6 Glenrath Hen Litter

Summary and Conclusion

Hen litter from Glenrath was treated with Xergi Nix technology and analyzed for biomethane potential and nitrogen content before and after treatment. Results from untreated hen litter is compared with Nix treated litter to evaluate the effect of the treatment.

BMP assays were initiated January 24th 2011 and run for 79 days.

	Units	Untreated Hen Litter	Treated Hen Litter
BMP	NmL CH_4/g VS (NmL CH_4/g wet weight)	322 (± 5) (51)	329 (± 9) (53)
Total Ammonium Nitrogen	N/kg VS	9,8 g (± 0,3)	5,5 g (± 2,3)
Total Kjeldahl Nitrogen	N/kg VS	82,5 (± 2,1)	77,0 (± 1,9)
Total Organic Nitrogen	N/kg VS	72,8 (± 2,1)	71,5 (± 3,0)
Total Solids content	w/w %	23,1	
Volatile solids content	w/w %	16,2	

Table 14 – Overview of key results associated with the analysis of hen litter.

- Treating the Hen litter with Nix technology had no observable effect on BMP.
- More than 90% of the final BMP yield was obtained after 14 days of incubation.
- Nix treatment had no effect on removal of organic nitrogen, however ammonium/ammonia levels are decreased by 40-50%.
- Total nitrogen levels were decreased by 7% due to Nix treatment.

Results

TS and VS analysis

TS and VS contents of the hen litter were determined in triplicate prior to Nix treatment.

	Unit	Untreated Hen litter	
Total Solids	w/w%	23,1	± 0,05
Volatile Solids	w/w%	16,2	± 0,23

Table 15 – VS and TS levels of hen litter

Figure 21 – Hen Litter

BMP analysis

Specific methane yields obtained during the BMP analysis of treated and untreated hen litter are shown in Figure 22. All curves show a steady increase in gas yield until a maximum yield is obtained on day 23 after which the substrates no longer contribute to methane production. Error bars represent ±1 standard deviation.

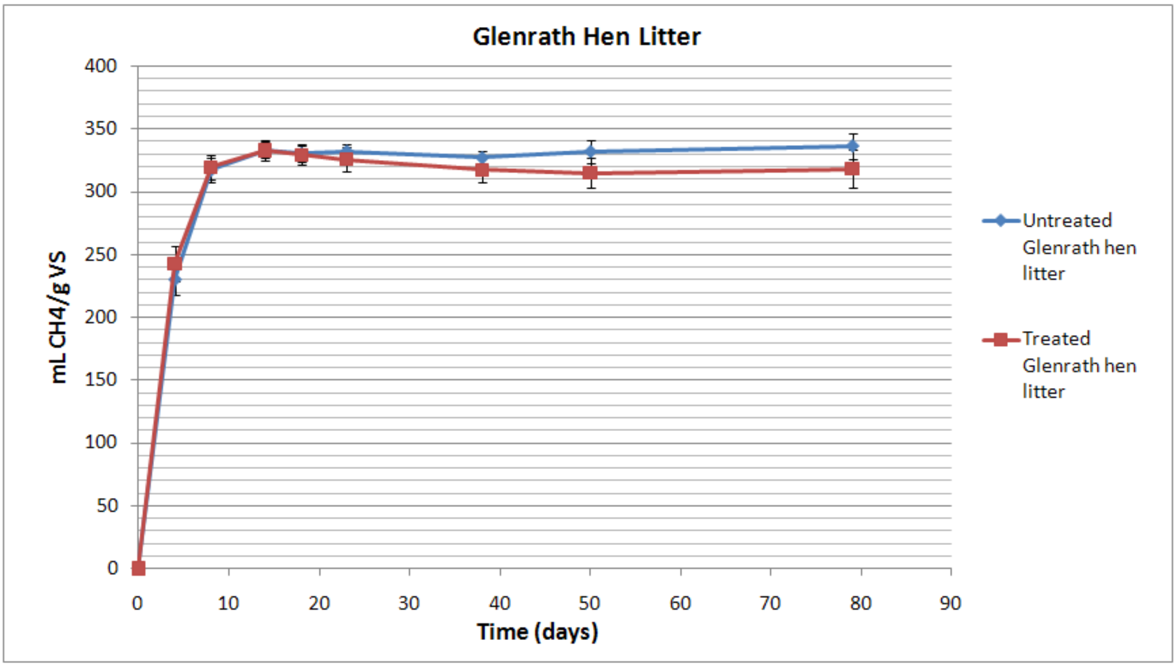


Figure 22 - Methane yield from batch bottles with treated and untreated hen litter.

After 79 days data for maximum yield show BMP values of 322 (± 5) NmL CH_4/g VS for the untreated hen litter and 329 (± 9) NmL CH_4/g VS for treated hen litter. On day 14 more than 90% of the final yield had been obtained.

Based on a kinetic analysis of the gas production the expected yield in a full scale continuous AD plant with a 20 days thermophilic primary digestion and a 10 days mesophilic secondary digestion will be approx. 290 NmL CH4/g VS equal to 90% of the obtained batch yield.

Nitrogen analysis
The nitrogen content of treated and untreated hen litter are shown in Figure 23. Specific values and the resulting nitrogen reductions as a result of Nix treatment are shown in Table 16.

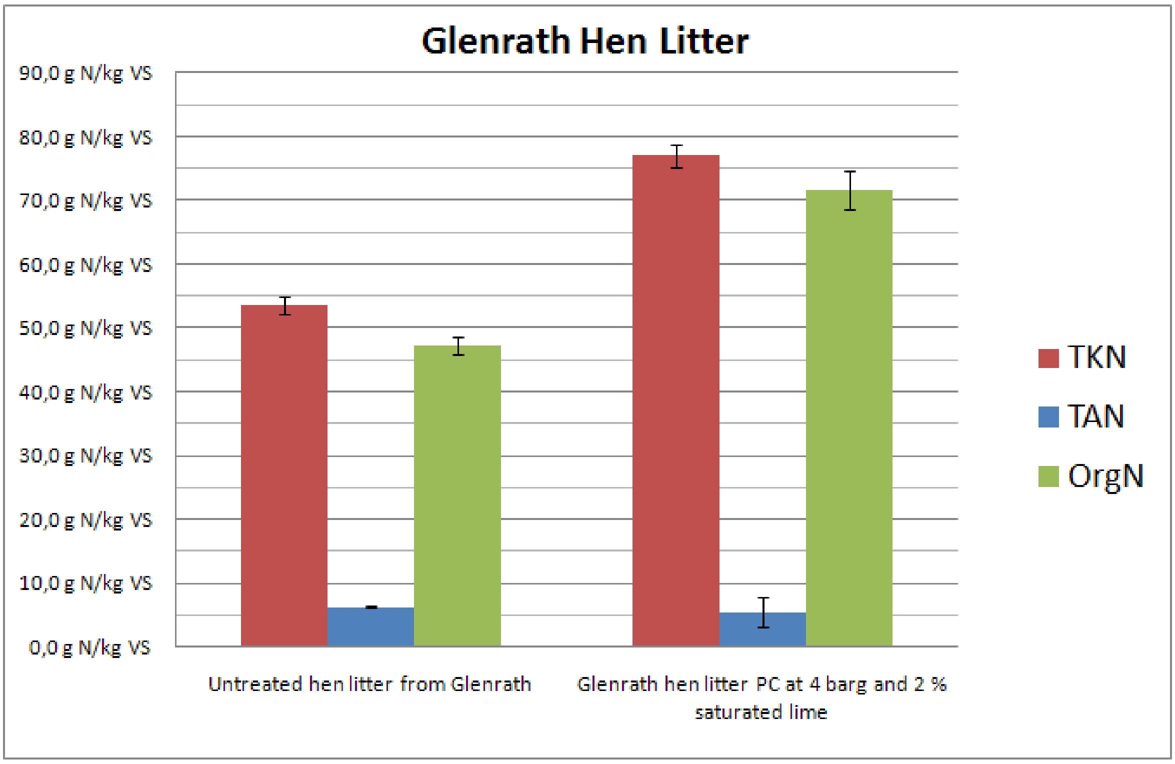


Figure 23 – Graphical representation of TKN, TAN and Organic nitrogen (Org-N) levels in untreated and treated hen litter. Org-N is calculated from TKN and TAN levels (Org-N = TKN – TAN).

Total ammonium nitrogen content	TAN **(g N/kg VS)**	SD **(g N/kg VS)**	RSD %	Reduction **(g N/kg VS)**	Reduction in percent
Untreated hen litter from Glenrath	9,8	0,3	3%		
Treated hen litter	5,5	2,3	43%	4,3	44%

Total kjeldahl nitrogen content	**TKN (g N/kg VS)**	**SD (g N/kg VS)**	**RSD %**	**Reduction (g N/kg VS)**	**Reduction in percent**
Untreated hen litter from Glenrath	82,5	2,1	3%		
Treated hen litter	77,0	1,9	2%	5,5	7%

Total organic nitrogen content	**Org-N (g N/kg VS)**	**SD (g N/kg VS)**	**RSD %**	**Reduction (g N/kg VS)**	**Reduction in percent**
Untreated hen litter from Glenrath	72,8	2,1	3%		
Treated hen litter	71,5	3,0	4%	1,2	2%

Table 16 – Values of TAN, TKN and Org-N with standard deviations (SD), relative standard deviations (RSD) and reductions as a consequence of Nix treatment.

Inorganic nitrogen constitutets a rather small amount of the entire nitrogen pool (~12%). Organic nitrogen constitutes the remaining ~88%. The measured removal of inorganic nitogen is therefore somewhat uncertain, as a small release of nitrogen from the organic N pool after the Nix treatment potentially could blur the measurement significantly. Generally the removal of inorganic nitrogen from other biomasses is 60 – 70 % at the described treatment conditions.

5 CSTR investigations

5.1 Introduction

The project was run at the experimental facilities of Xergi in Foulum (Denmark) and it included two parts:

Part 1: lab-scale tests (batch);
Part 2: pilot-scale tests (CSTR);

This part of the report focuses on Part 2, the pilot-scale CSTR tests. The tests lasted for 9 months, from September 2010 to May 2011:

Sep. 2010 – Dec. 2010:	thermophilic operation (phase 1A, phase 1B)
Jan. 2011 – May 2011:	mesophilic operation (phase 2)

5.1.1 Objective of the project

The objective of the project was to run a stable biogas process fed with chicken litter monosubstrate and with minimal water addition. Also, the ammonia removal with NiX treatment had to be determined.

Nitrogen is required by the microorganisms and often the nitrogen source is ammonia NH_3.However, NH_3 can be toxic. Chicken litter contains high amount of nitrogen (mainly organic nitrogen) and this results in high NH4-N concentration in the biogas digester (ammonium nitrogen, sum of ammonia NH_3 and ammonium ions NH_4^+). Also, chicken litter has high TS content and to be pumped it requires dilution.

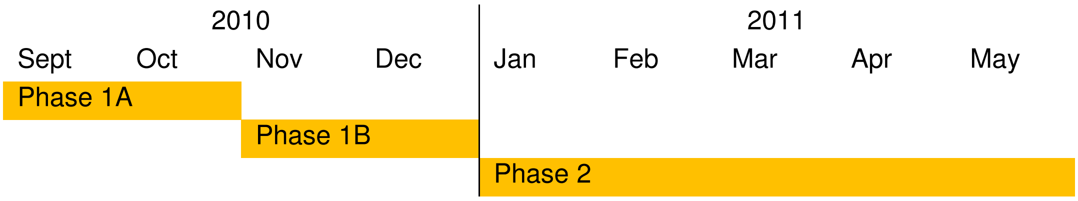
5.1.2 Choice of thermo-chemical treatment (NiX)

The treatment conditions used for the thermo-chemical treatment was based on the patented NiX treatment and on the results described in the first part of the report (Part 1). Details of the treatment conditions used during Part 2 (described in this part of the report) are given in Appendix A: NiX treatment.

5.2 Procedure

5.2.1 CSTR experiments

Xergi's pilot-scale plant *Anlæg 1* was used for the experiments. A 1-step CSTR biogas process was run at thermophilic conditions (52 °C) during the first period of the experiment until December 2010 (Phase 1A, Phase 1B) and at mesophilic conditions (37 °C) from January 2011 until May 2011 (Phase 2). Chicken litter was the substrate used.



Sept 2010 – Oct 2010: phase 1A, thermophilic operation no NiX
Nov – Dec 2010: phase 1B, thermophilic operation with NiX
Jan 2011 – May 2011: phase 2, mesophilic operation

Mesophilic conditions were chosen to minimize the negative effect of high NH4-N concentration in the digester.

5.2.2 Phase 1: Thermophilic conditions

5.2.2.1 Substrate

The substrate was a mixture of raw chicken litter and water at a ratio of 1:4.17 (1 kg wet weight of chicken litter and 4.17 kg of water). The chicken litter was obtained from a local farm (Tjele, Denmark) breeding chickens (broilers). Chicken litter was collected on August 24 2010, distributed into barrels (approximately 60 kg each) and frozen at -10 °C. The chicken litter contained approximately 2.3% of sphagnum wet weight. The chickens were 42 days old when the chicken litter was collected.

The raw chicken litter contained TS 62.8 ± 1.8%, VS 51.2 ± 2.7% (average of 11 measurements), NH4-N 7.0 ± 0.7 g/kg (average of 15 measurements), TKN 28.8 ± 1.4 g/kg (average of 9 measurements).

5.2.2.2 Phase 1A: thermophilic, without NiX treatment

Raw chicken litter and water in the proportions above were used as substrate (Figure) from September 2010 until November 1 2010. The organic loading rate (OLR) was maintained at 6.0 ± 1.0 kg VS / (m^3 d), where VS is the organic matter content of the raw chicken litter. The hydraulic retention time (HRT) was 14.6 ± 2.0 d.

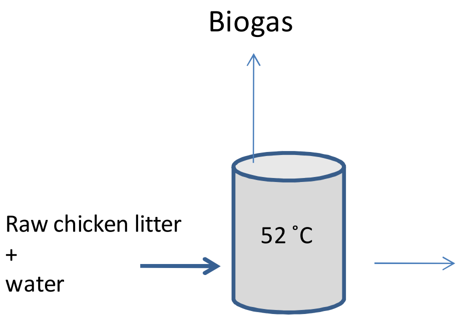


Figure 24. Process configuration during thermophilic operation without NiX treatment

5.2.2.3 Phase 1B: thermophilic, with NiX treatment

Between November 2 2010 and December 2010, the substrate was made of raw chicken litter and water in the proportions above treated with NiX (Figure 1). Details of NiX treatment are given in Appendix A: NiX treatment. The organic loading rate (OLR) was maintained at 6.0 ± 1.0 kg-VS / (m^3 d), where VS is the organic matter content of the raw chicken litter. The hydraulic retention time (HRT) was 14.6 ± 2.0 d.

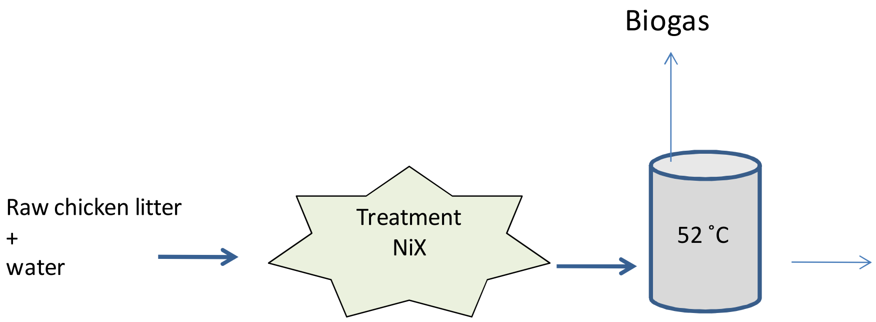


Figure 1. Process configuration during thermophilic operation with NiX treatment

5.2.3 Phase 2: mesophilic conditions

5.2.3.1 Substrate

The substrate was a mixture of chicken litter and liquid fraction separated from the effluent of the biogas process. The mixture was treated with NiX before being used as substrate. No water was added to the substrate (aside from steam added during NiX treatment, approximately 0.05 kg-H_2O / kg WW chicken litter).

5.2.3.2 Mesophilic, re-circulation, no water addition

Re-circulation of the liquid fraction of the effluent was applied (Figure 25). The OLR was 4.4 ± 0.3 kg VS / (m3 d).

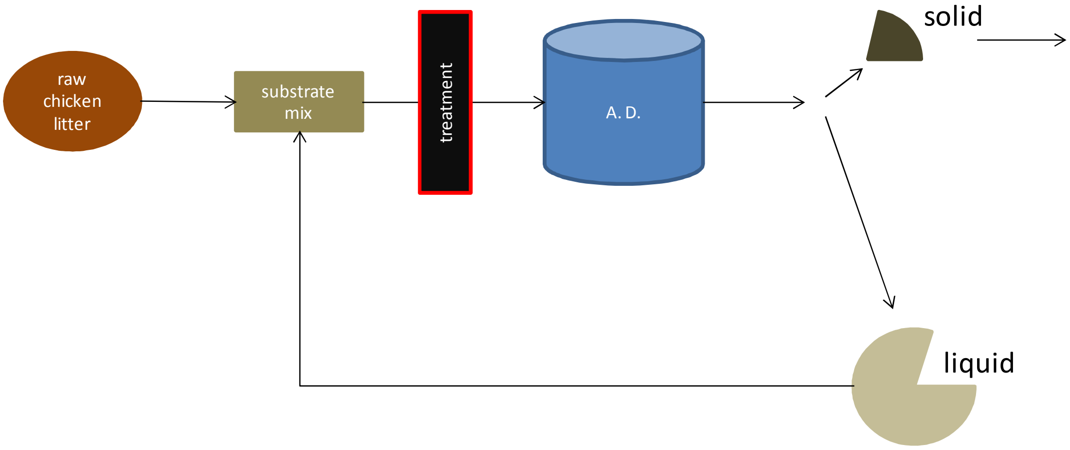


Figure 25. Process configuration during phase 2 (mesophilic operation with re-circulation and NiX treatment)

5.2.4 Batch tests

The substrates for the batch tests were sampled during the CSTR experiments. Details about the preparation of the batches are given in Appendix B: batch experiments.

5.3 Results

Treatment with NiX removed 47% of the NH4-N from the substrate. With NH4-N concentration higher than 6.4 g / kg WW in the digester, the methane yield showed clear signs of decrease. Addition of water was required for stable operation of the CSTR. Batch tests resulted in 13% higher methane yield from NiX-treated chicken litter compared to raw chicken litter.

5.3.1 Effect of NiX treatment

5.3.1.1 Effect of NiX treatment on NH4-N concentration in the digester

The effect of NiX treatment on the NH4-N concentration in the digester was tested during thermophilic operation.

The NH4-N concentration in the digester decreased from 3.30 ± 0.14 to 2.79 ± 0.24 g / kg WW (Figure26).

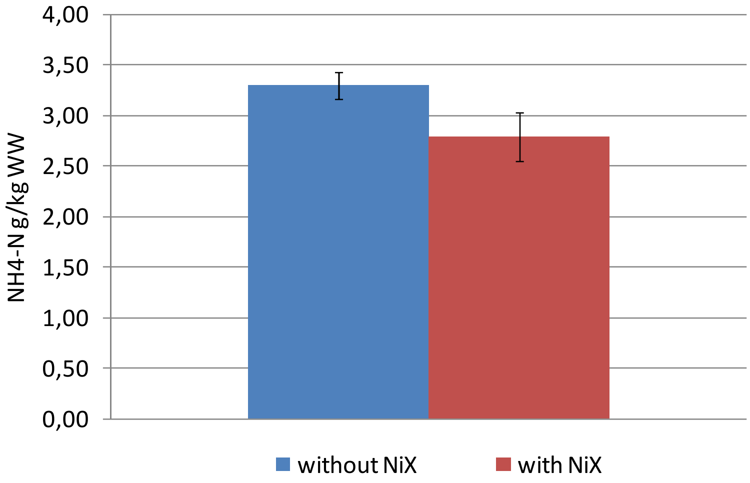


Figure 26. Effect of NiX treatment on NH4-N concentration in the digester

5.3.1.2 Effect of NiX treatment on NH4-N concentration in the substrate

The effect of NiX treatment on the concentration of NH4-N in the substrate was studied during mesophilic operation with accurate mass balances.

NiX treatment removed 47% of the NH4-N from the substrate.

The rate of pressure reduction after NiX treatment could be regulated by adjusting the opening of the valve for pressure release. This affected the amount of loss, because increasing the rate of pressure reduction increased the amount of material that leaved the NiX treatment unit and thereby was “lost”. The optimal rate of pressure release had to maximize NH4-N removal and the amount of treated substrate collected after the treatment (i.e. the loss had to be minimized). Also, the time needed for the NiX treatment had to be minimized.

Avoiding losses, 47% NH4-N removal was obtained. Increasing the rate of pressure release and allowing part of the treated material to be lost (4 kg WW), removed 60% NH4-N (corrected for the mass loss), (Figure).

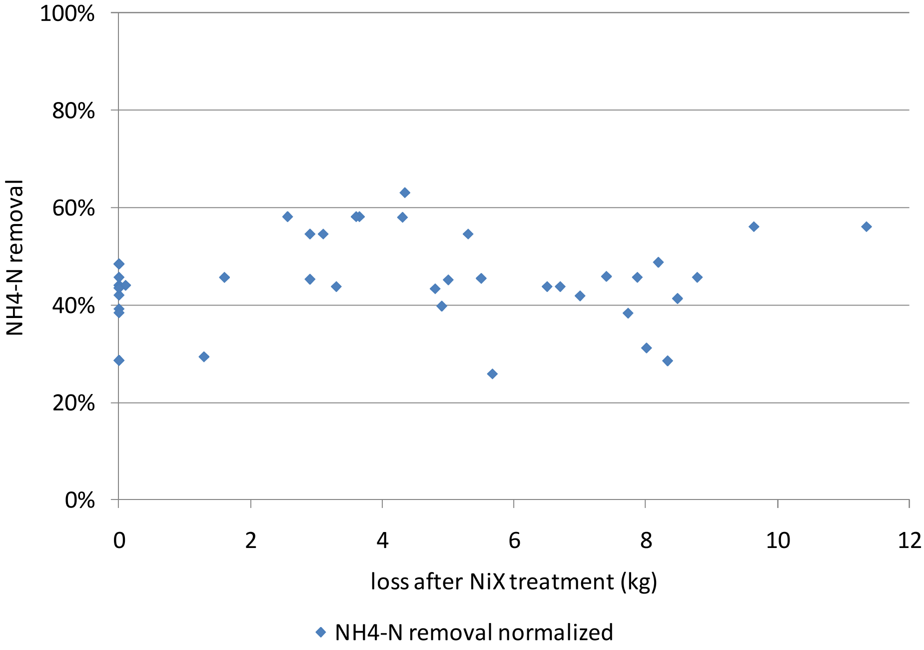


Figure 27. Removal of NH4-N with NiX treatment vs. loss caused by high rate of pressure reduction

Based on the results shown in Figure , it was chosen to operate the NiX treatment unit avoiding losses to minimize the amount of material to be treated with NiX and to make the mass balance easier.

5.3.1.3 Effect of NiX treatment on yield

The effect of NiX treatment on the yield of chicken litter was measured with batch tests. The substrates used for the batch tests were the same as those used for the CSTR experiments.

The batch tests resulted in 303 ± 5 mL CH_4 / g VS (where VS is the organic matter content of the raw chicken litter) and showed that NiX treatment increased by 13% the methane yield of chicken litter (on VS basis, contribution from the re-circulated liquid is subtracted), Table 17.

Table 17. Effect of NiX on the methane yield of the chicken litter

Yield chicken litter	L CH_4 / kg VS
Without NiX treatment	303
With NiX treatment	343
increase	13%

Details about the preparation of the batches are given in Appendix B: batch experiments.

The comparison of the results described in this report (Part 2 of the project) to the results in the first part of the report (Part 1 of the project) shows that a higher yield improvement was achieved in Part 1 (22% improvement). The difference can be explained with differences in the raw chicken litter used during the two parts of the project. The raw chicken litter used for the experiments in Part 1 was not from the same batch used for the experiments in Part 2, therefore the effect on Nix may have been different.

The fact that the raw chicken litter of Part 1 was different from the one of Part 2 is confirmed by the different methane yields: 277 L CH_4 / kg VS (Phase 1) and 303 L CH_4 / kg VS (Phase 2). Also, the smaller-scale of the experiments of Part 1 resulted in higher NH4-N removal (65% NH4-N removal). This shows that the amount of raw material to be treated affects the effect of NiX. With low amounts, raw samples are homogenous and have low mass compared to the headspace and the effect of NiX can be maximised. Finally, the experiments of Phase 1 were made at 10% TS, while the substrate treated during Phase 2 contained approximately 22% TS.

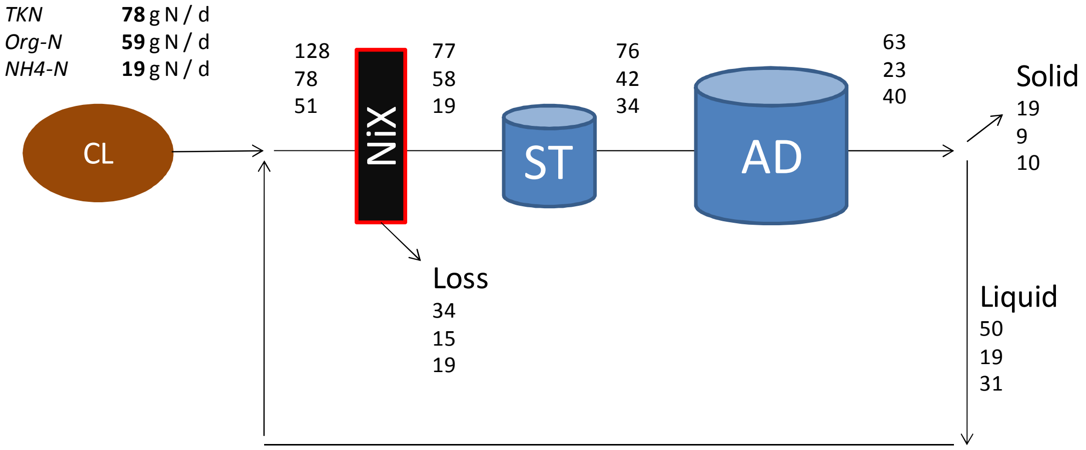
5.3.1.4 Effect on nitrogen degradation

Nitrogen flow and degradation in the system

Nitrogen measurements (NH4-N and TKN) were made once per week during the mesophilic operation (Phase 2). The concentration of organic nitrogen was calculated as the difference between TKN and NH4-N.

Uric acid was measured once on May 10 2011. Because the results from uric acid measurements in this report are preliminary, they are described only in Appendix C: uric acid.

In Figure 28, the nitrogen flow in the system is expressed in g of nitrogen per day. This flow is calculated from the measured concentrations (g nitrogen per kg wet weight, measured once per week) and the measured flow rates (kg wet weight per day, measured every day). The averages of two or three nitrogen measurements and of four or five flow rate measurements have been used to calculate the nitrogen flow.



CL = chicken litter raw
Liquid = liquid fraction of effluent digester after separation with centrifuge
NiX = NiX-treated material collected immediately after NiX treatment
loss = loss after NiX treatment during quick pressure release
ST = substrate tank
AD = biogas digester (anaerobic digester)
Solid = solid fraction of effluent digester after separation with centrifuge

Figure 28. Nitrogen flow in the system (data are flow rates, g of nitrogen per day)

Because the flows in Figure 28 are calculated from measurements, measurement errors and uncertainties have to be taken into account. For example, the balance around the NiX treatment unit shows that approximately 6% of organic nitrogen is missing:

58 + 15 = 73 g N / d
73 = 94% of 78 g N / d

This difference is probably due to measurement uncertainties.

Also, the system was not at steady state when the nitrogen flow was made. Therefore, exact balance between the nitrogen entering and leaving the system should not be expected.

Nitrogen balance substrate tank

The balance around the substrate tank is closed, as 77 g N / d (as TKN) enter the substrate tank and 76 g N / d leave it.

In the substrate tank, approximately 30% of the organic nitrogen was converted into NH4-N. This was probably due to microbial activity in the substrate tank (although the substrate tank was maintained at 10-15 ℃), as revealed by dedicated experiments. As a consequence of the 30% conversion of organic nitrogen into NH4-N, the substrate entering the biogas digester had higher NH4-N concentration compared to the material entering the substrate tank. This contributed to increase the NH4-N concentration in the digester.

A better understanding of the nitrogen degradation in the substrate tank is needed to allow development of methods to reduce the conversion of organic nitrogen into NH4-N or to remove the NH4-N from the substrate tank. This would result in lower NH4-N concentration in the biogas digester.

Nitrogen balance biogas digester

The TKN balance around the biogas digester shows that 17% TKN is missing: 76 g N / d enter the biogas digester with the substrate and only 63 g N / d leave it with the effluent. The main reason for this is the transient state of the biogas digester at the time of the measurements.

In the biogas digester, approximately 45% of the total organic nitrogen entering the digester was converted into NH4-N. Approximately 80% of the total organic nitrogen entering the digester was from the raw chicken litter, while the remaining 20% was from the re-circulated liquid used to prepare the mixture treated with NiX.

Nitrogen balance separation liquid-solid

A centrifuge was used to separate the effluent of the biogas digester into the liquid and the solid fraction. The TKN balance around the separation was accurate at 10%, which can be considered satisfactory.

On wet weight basis, approximately 70% of the TKN of the effluent was collected in the liquid fraction. More than 75% of the NH4-N of the effluent is collected in the liquid fraction.

Balance NiX treatment unit

The TKN and NH4-N in the fraction leaving the NiX treatment unit as gas during pressure release was not measured. The balance is based on the assumption that only steam and NH4-N were in this fraction (Figure 29, below).

Correcting for the mass loss on VS basis, NiX treatment removed 47% of the NH4-N, in average (**Figure** 27, above).

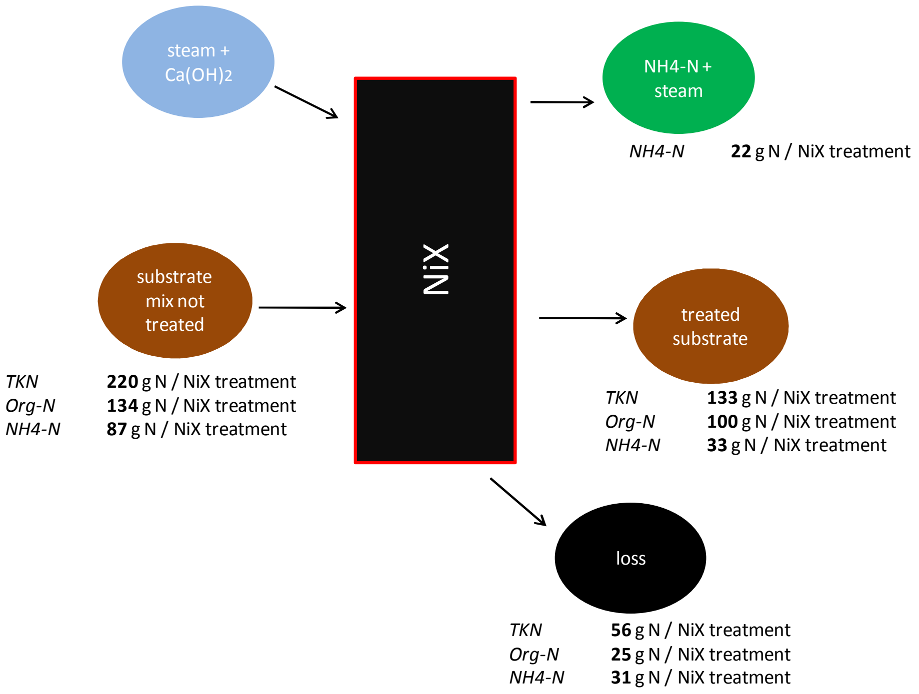


Figure 29. Nitrogen flow around the NiX treatment unit (data are in g of nitrogen at a specific NiX treatment)

Organic nitrogen was not affected by the NiX treatment or only a very small part of it was converted into NH4-N. Only 7% of the organic nitrogen was missing from the balance and this was within the accuracy of the measurements.

5.3.2 Effect of re-circulation

Re-circulation had the overall effect of increasing the TS content of the digester at the rate of 0.4% per week and increasing the NH4-N concentration of the digester at a rate of 0.5 g / kg WW per week. This was due to the lack of water (no water was added to the system, aside from steam during NiX treatment). In the substrate, the VS content was approximately constantly 68% of the TS (therefore the ashes were 32% of the TS). The VS fraction of the TS of the digester increased from 44% in February 2011 to 55% at the end of May 2011, indicating that the conversion of VS into biogas was decreasing. The increase was approximately linear. A similar increase was observed in the liquid fraction.

5.3.2.1 HRT and SRT

The HRT was different from the SRT (solids retention time, retention time of the alive microorganisms), Figure 30.

The HRT was calculated from the flow rate of material leaving the system. The HRT was approximately 145 d. This is the average time that solids and water spend in the system (the system includes the re-circulation). The HRT did not depend on the flow rate of the recirculation.

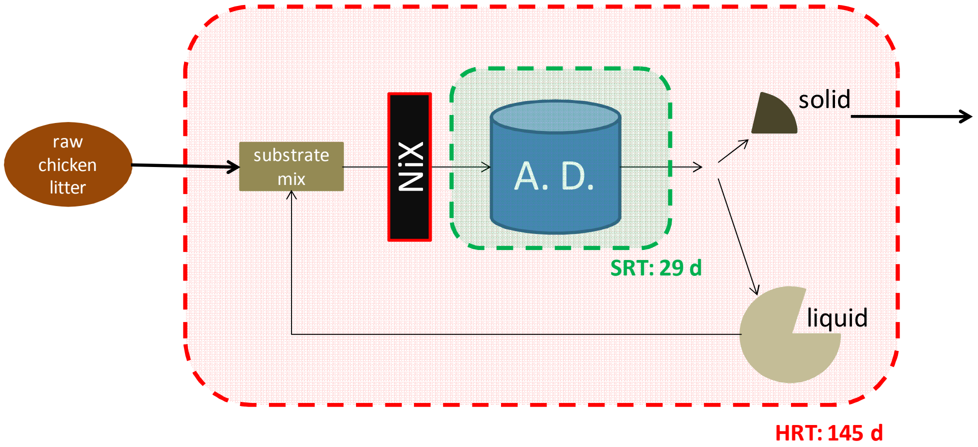


Figure 30. HRT and SRT

The SRT was calculated as the average time that the living microorganisms spend in the system. The SRT was 29 ± 5 d. This is acceptable for a mesophilic biogas process. The liquid fraction (re-circulate) contains microorganisms and these are killed during NiX treatment. Therefore, the retention time of the microorganisms (SRT) had to be calculated from the flow rate of the material that leaved the digester. The SRT depends on the flow rate of the recirculation.

5.3.2.2 Effect of recirculation on NH4-N and yield

During mesophilic operation, the yield reached the constant 297 ± 35 mL CH_4 / g VS (where VS is the organic matter content of the raw chicken litter).

The system showed clear signs of decreasing efficiency of conversion of substrate into methane starting from May 10 (Figure 31). The yield decreased strongly from 297 ± 35 mL CH_4 / g VS with a rate of approximately 8 mL CH_4 / g VS per day. This was due to high NH4-N concentration in the digester (Figure 31, Figure 32, Figure33).

On May 10, the NH4-N concentration in the digester was 6.4 g / kg WW and this can be considered the threshold level that caused decreased conversion efficiency or inhibition. On May 10, the concentration of CH_4 in the biogas was 55% and the tot VFA in the digester was 2000 mg/L. On May 23, the tot VFA in the digester was 2400 mg/L, just slightly higher than on 10 May. The methane yield was decreasing, but the tot VFA concentration did not show any clear or sharp increase.

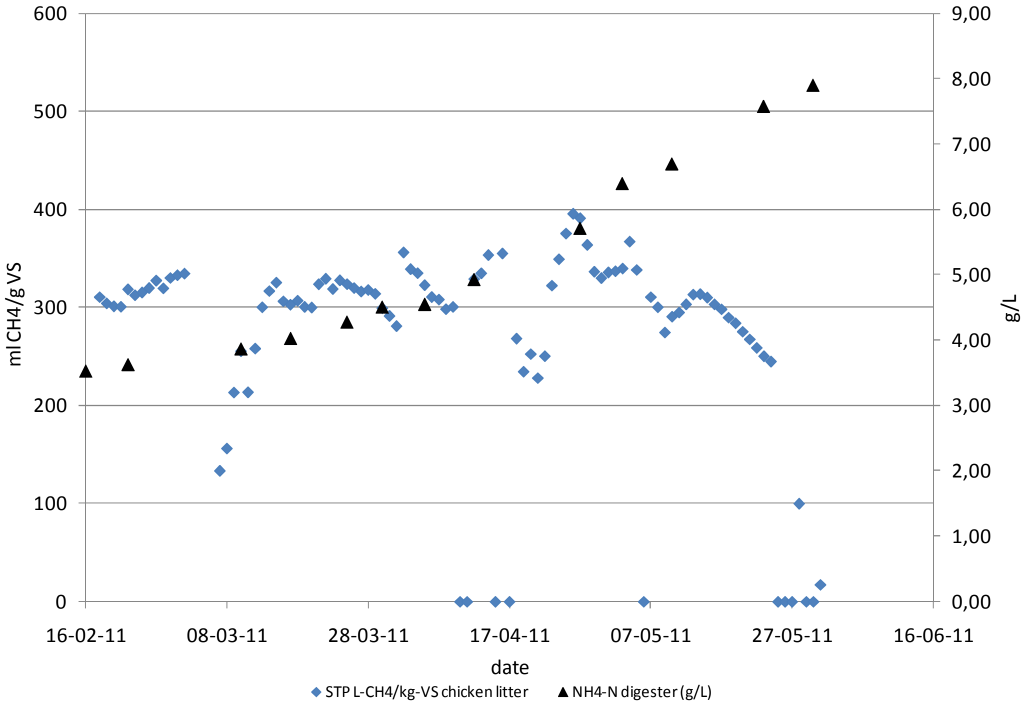


Figure 31. Methane yield during phase 2 (mesophilic operation)

The methane yield decrease of March 3 – March 7 was due to a feeding pump stop. In the period April 13 – May 7 the yield showed large variations (the points in Figure 31 are scattered) because of technical problems with pumps and with the computer controlling the pilot plant.

The TS and NH4-N concentration in the digester always showed increasing trends (Figure 32, Figure33).

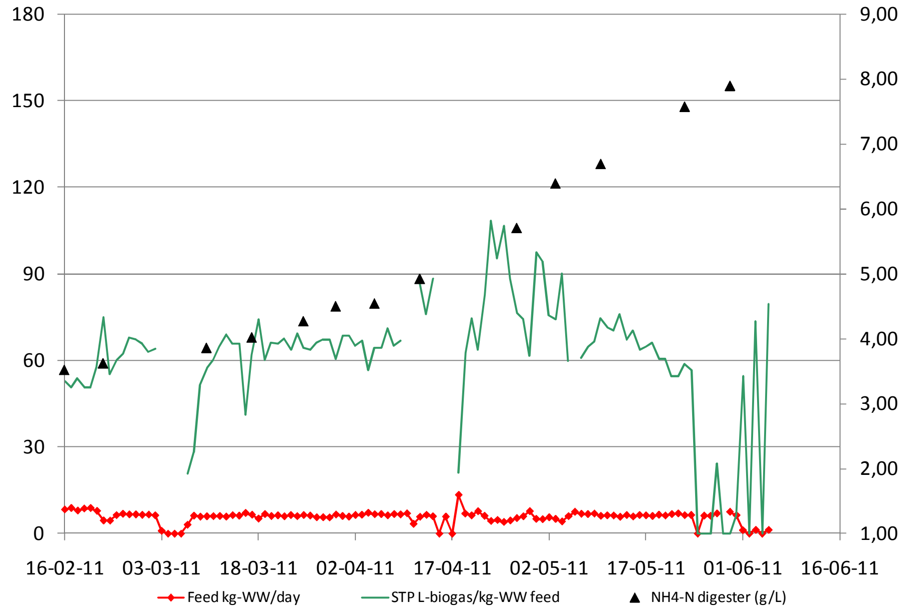


Figure 32. Feed flow rate, biogas yield (main vertical axis), NH4-N concentration in the digester (secondary vertical axis).

5.3.2.3 Effect of re-circulation on separation of liquid fraction

The effluent of the digester was separated into the liquid and the solid fraction. The liquid fraction was re-circulated and mixed with the raw chicken litter before being treated with NiX.

The separation was made by sedimentation until middle of March 2011 and with a centrifuge from middle of March 2011 and can be seen in Figure33. The new separation method (centrifugation) was introduced to decrease the TS% of the liquid fraction, because sedimentation became not effective as the TS% of the material to be separated increased. The purpose was to decrease the TS% in the digester by decreasing the TS% of the separated liquid. However, lower TS% in the liquid obtained with centrifugation was not sufficient to lower the TS% in the digester.

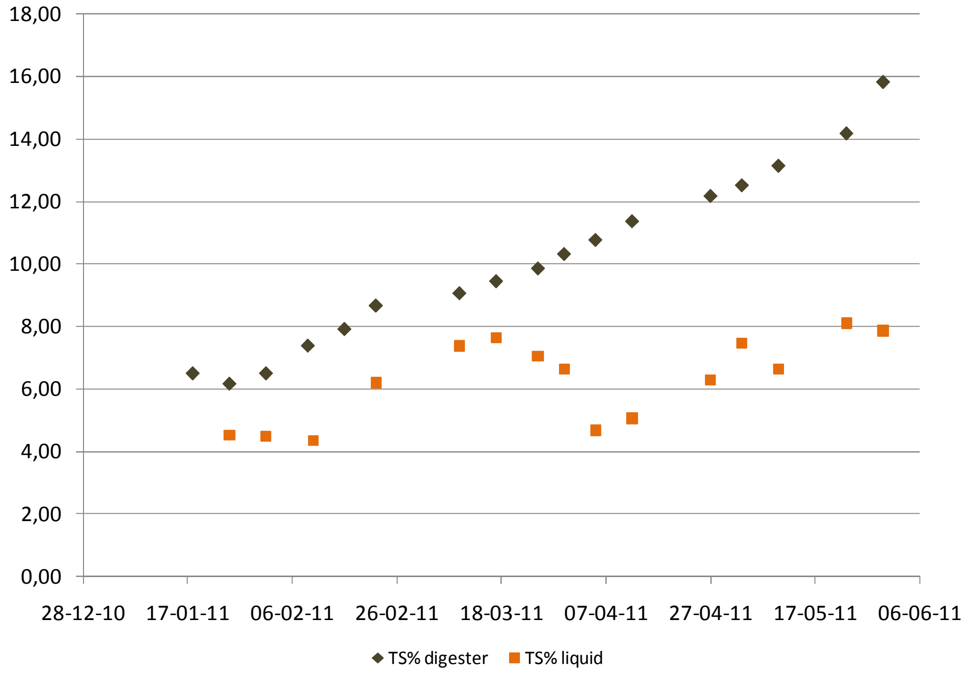


Figure 33. TS content of the digester and of the liquid after separation with centrifuge.

Approximately 75% WW was collected in the liquid fraction. The TS content of the liquid fraction increased as the TS content of the digester effluent increased, i.e. the separation efficiency of the centrifuge used for separation depended on the TS content of the material to be centrifuged.

5.3.2.4 Effect of re-circulation on water requirements

A very low amount of water was added to the system. This was done adding steam during NiX treatment.

Steam was added to the substrate during NiX treatment and was released from it at the end of the treatment, when the pressure was released. The net amount of water added to the system during NiX treatment was the difference between the steam entering the substrate (approximately 0.1 kg steam/kg-chicken litter, according to the steam flow meter) and the steam leaving the treated substrate. The net amount of water added during NiX treatment was measured as the difference of water in the untreated substrate (water in the raw chicken litter plus water in the re-circulated liquid) and the water in the treated substrate. On average, 0.05 kg of water (condensed steam) for each kg of raw chicken litter entered the system during NiX treatment.

Water was leaving the system via the solid fraction after separation. Mass balances and experimental data showed that the amount of water leaving the system was higher than the amount of water entering it.

5.3.3 Balances NiX treatment

Figure 34 shows the mass balance around the NiX treatment unit for a specific treatment. The nitrogen balance (NH4-N) showed that NiX treatment removed 55% of NH4-N from the substrate. The concentration of NH4-N in the treated substrate (after NiX treatment) was 3.2 g-NH4-N/kg-WW. These data are from one specific treatment considered. On average, NiX treatment removed 47% NH4-N.

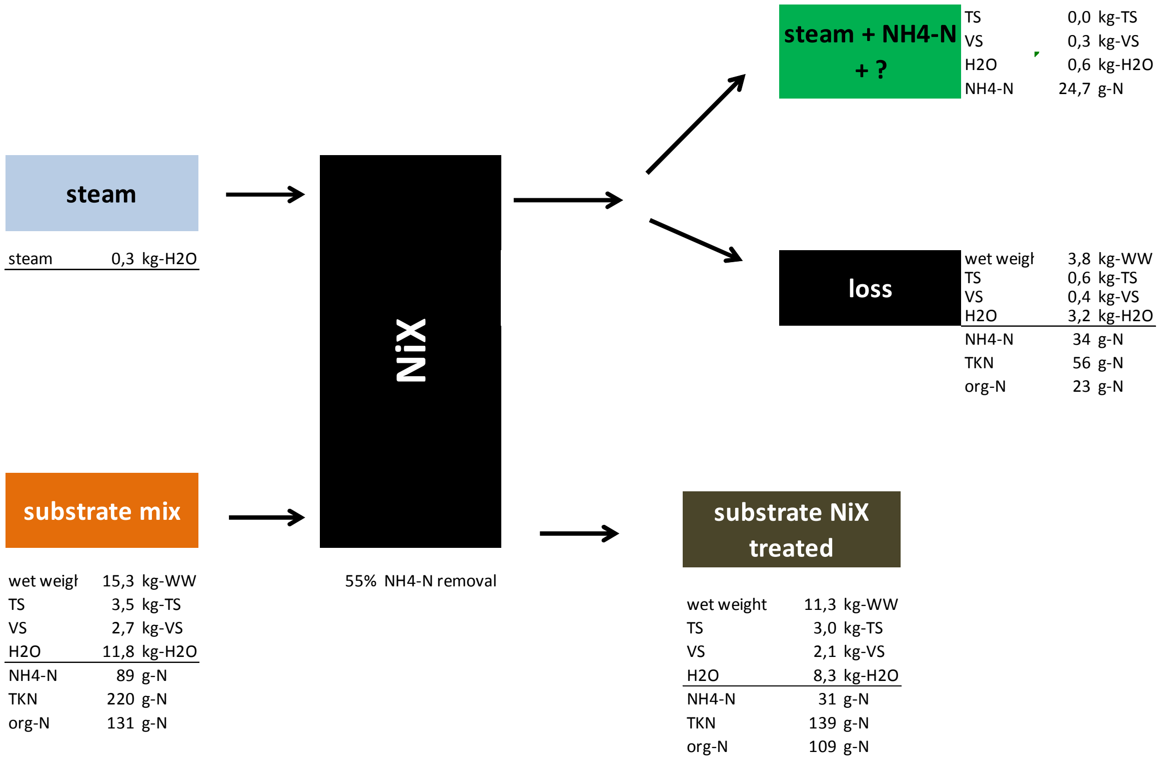


Figure 34. Mass balance NiX treatment (data are per treatment)

5.4 Conclusions

NiX treatment removed 47% of the NH4-N from the substrate and decreased by 15% the NH4-N concentration in the biogas digester.

Re-circulation of liquid fraction separated from the effluent of the biogas digester caused increasing TS and NH4-N concentration in the digester because of lack of water. Addition of extra water (besides 0.05 kg H_2O / kg WW chicken litter as steam during NiX treatment) is needed to run stable biogas processes based on chicken litter as monosubstrate.

The methane yield was stable at 297 ± 35 mL CH_4 / g VS (where VS is the organic matter content of the chicken litter) and decreased probably because of NH4-N inhibition when the NH4-N concentration in the digester became higher than 6.4 g / kg WW.

Nitrogen measurements showed that the concentration of organic nitrogen does not change during the 20-minute NiX treatment. Approximately 30% of the organic nitrogen is converted into NH4-N during the storage of the NiX treated mixture in the substrate tank. The biogas process resulted in 45% conversion of organic nitrogen into NH4-N.

5.5 Appendices

5.5.1 Appendix A: NiX treatment

NiX treatment was applied with Xergi's thermo-chemical treatment unit. The substrate for NiX treatment were the mixtures of raw chicken litter and water or of raw chicken litter and liquid separated from the digester's effluent.

Steam at 8-12 bar was injected into a chamber containing the substrate in the proportions given below. When the set-point relative pressure (4 bar, corresponding to approximately 140 ℃ inside the chamber) was reached, it was maintained for 20 minutes. The sample was mixed during the treatment.

Pressure is expressed as relative pressure above atmospheric pressure (gauge pressure).

A pre-heating of the external walls of the chamber and injection of steam on these walls during NiX treatment was made to minimize steam condensation inside the chamber during the treatment. At the end of the treatment, the pressure was released in approximately 2 hours. The treatment conditions are summarized below:

Thermophilic operation:
Sample: raw chicken litter
Water: 4.17 kg water per kg WW of raw chicken litter
Catalyst: 2% $Ca(OH)_2$ (percentage by wet weight of total mixture)
Treatment: 4 bar (140 ℃), 20 min, mixing

Mesophilic operation:
Sample: raw chicken litter
Water: no water added, aside from steam during steam treatment (net approximately 0.05 kg steam per kg WW chicken litter)
Liquid: liquid fraction from effluent biogas digester, 2.61 kg WW / kg WW raw chicken litter
Catalyst: 2% $Ca(OH)_2$ (percentage by wet weight of total mixture)
Treatment: 4 bar (140 ℃), 20 min, mixing

5.5.2 Appendix B: batch experiments

The methane potential was determined in batches, with infusion bottles of 543 mL total volume, at 52 ℃. Anaerobic digested effluent from a thermophilic biogas plant (52 ℃) was used as inoculum for the batch assays.

Inoculum (200 mL) and substrate were added into the bottle in the amounts given in Table 19. Control batches with pure cellulose as substrate (for examination of inoculum viability) and blank batches without substrate (to measure the methane production from the inoculum) were included. The assays were made in triplicates.

To calculate the effect of NiX on the methane yield of the chicken litter, the results from the batch tests were corrected for the ratio between raw chicken litter and total mixture and for the contribution to the methane yield from the liquid separated from the effluent. The results of the calculations are given in Table **18**18:

Table 18. Effect of NiX on the methane yield of the chicken litter

Yield chicken litter	L CH4 / kg VS
Without NiX treatment	303
With NiX treatment	343
Increase	13%

Table 19. Batch experiments: batch preparation and methane yield

Date batch start	Name batch	Substrate WW (g)	Substrate TS% (%)	Substrate VS% (%)	Methane yield (L-CH_4 / kg-VS)
May 03 2011	Raw chicken litter	2.87	62.6	52.1	303 ± 8
	Liquid	16.41	6.3	3.4	121 ± 6
	Substrate collected immediately after NiX treatment	6.80	21.7	16.6	314 ± 18

5.5.3 Appendix C: uric acid

Uric acid was measured once as a preliminary test. Uric acid measurements were made to have a better understanding of the degradation of nitrogen in the system.

Uric acid is a non-protein organic nitrogen compound, main end product of purine metabolism and present mainly in urine.

On 10 May 2011, preliminary uric acid measurements were made on samples of:

- Raw chicken litter
- NiX-treated mixture of raw chicken litter and liquid fraction from effluent digester
- Material lost during pressure release from NiX treatment unit
- Substrate sampled from substrate tank
- Digester effluent
- Liquid fraction separated from digester effluent
- Solid fraction separated from digester effluent

The preparation of the samples was made as described in:

B. Pekič, B. Slavica, Z. Zekovič
High-Performance Liquid Chromatographic Determination of Uric Acid in Feces of Egg-Laying Hens
Chromatographia Vol. 27, No. 9/10, May 1989.

The dried sample was extracted into a solution of Li_2CO_3 (extraction made in Xergi's laboratory) then this liquid fraction was injected into a HPLC (HPLC analysis made by Aalborg university)
The results of the preliminary uric acid measurements are given in Table **20**20 (concentrations of TKN and NH4-N are given for comparison):

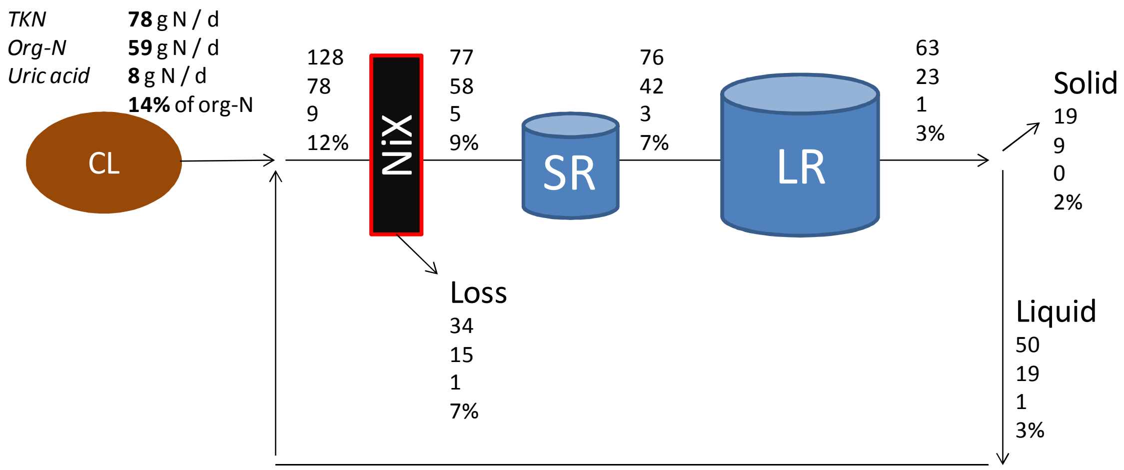
Table 20. Results from preliminary uric acid measurements (data are concentrations)

	Uric acid		TKN	NH4-N
	g uric acid / kg TS	g N / kg WW	g N / kg WW	g N / kg WW
Raw C. L.	15.44	3.10	30.37	7.24
NiX-treated	10.40	0.84	12.07	3.26
Loss	8.27	0.36	12.16	6.44
Substrate tank	5.84	0.42	12.29	5.42
Effluent digester	1.87	0.08	10.56	6.69
Liquid	3.26	0.07	8.58	5.35
Solid	0.89	0.10	11.82	5.95

According to the preliminary measurements for raw chicken litter, uric acid is approximately 10% of the TKN. This result is lower than values from literature, which indicate that uric acid is approximately 60% of total N.

The reason for this difference may be an incomplete extraction of uric acid from the sample into the liquid fraction to be injected into the HPLC. Results from HPLC seem reliable, according to the calibration curve.

The flow and degradation of uric acid in the system were calculated with the concentrations obtained from the preliminary uric acid measurements. These are summarized in Figure35:



CL = chicken litter raw
Liquid = liquid fraction of effluent digester after separation with centrifuge
NiX = NiX-treated material collected immediately after NiX treatment
loss = loss after NiX treatment during quick pressure release
SR = substrate tank (small reactor)
LR = biogas digester (large reactor)
Solid = solid fraction of effluent digester after separation with centrifuge

Figure 35. Nitrogen flow in the system (data are flowrates, g of nitrogen per day)

The data in Figure35 are flowrates, g of nitrogen per day. These have been calculated from the measured concentrations (g of nitrogen per kg wet weight) and the measured flowrates (kg wet weight per day). Therefore, measurement errors and uncertainties have to be taken into account when considering the flows shown in Figure5.

For example, the balance around the NiX treatment unit shows that approximately 6% of organic nitrogen is missing:
58 + 15 = 73 g N / d
73 = 94% of 78 g N / d
This difference is probably due to measurement uncertainties.

The degradation of uric acid was slightly higher than the degradation of organic nitrogen (Table 21), however a similar trend can be observed: lowest degradation during NiX treatment, higher in substrate tank and highest in biogas digester.

Table 21. Degradation of org-N and uric acid in the system

	Degradation org-N	Uric acid
During NiX	<5%	33%
In substrate tank	28%	40%
In biogas digester	45%	67%

The results above have to be considered preliminary because of the suspicion of incomplete extraction of uric acid during measurements.