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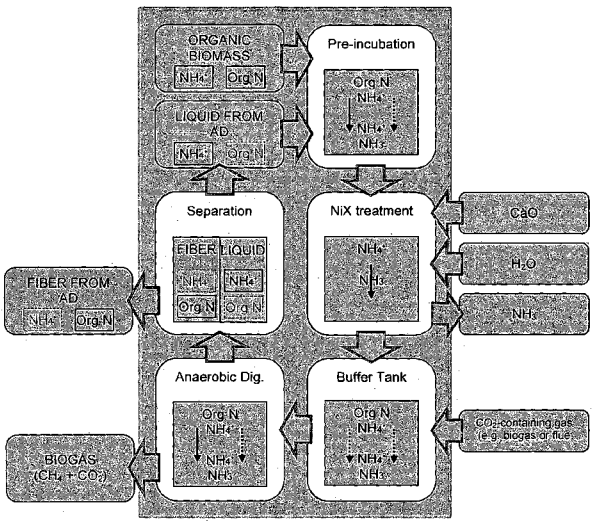
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(54) Title: METHOD FOR PH-CONTROLLED FERMENTATION AND BIOGAS PRODUCTION

(57) Abstract: The present invention is in the field of biomass processing and bioenergy production and facilitates efficient biomass processing and an increased production of renewable energy from processing and anaerobic fermentation of a wide variety of organic materials. In order to control the pH value of the biomass during processing thereof, a CO_2 containing gas, such as e.g. biogas or flue gas, is added to the biomass present in the buffer tank and/or in the anaerobic digester operably linked to the buffer tank.

Fig. 1



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Method for pH-controlled Fermentation and Biogas Production

All patent and non-patent references in the present application are hereby incorporated herein by reference in their entirety.

Technical Field of the Invention

The present invention is in the field of biomass processing and bioenergy production. In particular, the present invention aims to increase the amount of biogas one can produce when processing organic material.

Background of the Invention

The supplies of traditional energy sources, such as e.g. oil and coal, are finite and there are considerable concerns over the extent of remaining reserves. A continued utilization of fossil fuels as a dominant energy source is not consistent with the long-term sustainability of our environment.

Renewable energy sources represent a promising alternative to many traditional energy sources. Biomass represents one source of renewable energy. However, if biomass energy is to have a long-term, commercial future, the organic material must be processed to generate affordable, clean and efficient energy forms, such as liquid and gaseous fuels, or electricity.

Biomass processing remains important to ensure an efficient exploitation of the biomass energy. The energy potential can often be difficult to exploit and it can be present in a form which may only be exploited following extensive processing of the biomass. An increased exploitation of the energy potential of a biomass may result in an increased production of renewable energy sources, such as biogas.

One of the challenges associated with extraction of energy from biomass comprising organic materials is optimization of the energy yield from such a process. While the biomass itself may be perceived to contain an energy reservoir, this energy reservoir can not readily be released in a convenient form.

Accordingly, one challenge is to extract as much energy as possible from the biomass, by use of as little energy as possible, in order to increase the total energy yield of the process. The present invention aims to secure this objective.

Summary of the Invention

The present invention facilitates efficient biomass processing and an increased production of renewable energy from processing and anaerobic fermentation of a wide variety of organic materials.

Many types of organic materials have a high energy potential which can be exploited by processing the organic material. One form of processing an organic material is by performing an anaerobic fermentation resulting in the production of biogas. This process represents a conversion of an energy potential to a readily usable energy source.

Pre-treatment of biomasses - including lime pressure cooking - and partial stripping of ammonia N prior to performing a biogas fermentation, is not always sufficient to preclude an undesirable inhibition of biogas producing bacteria by ammonia released from organic bound N not stripped during the pre-treatment step.

Hence, there is a need for novel and innovative methods for removal of organic bound nitrogen (N) from different biomasses. In particular, there is a need for reducing costs and optimizing the consumption of energy used for biomass processing and in particular the removal of ammonia N from AD (anaerobic digestion) processes in which e.g. poultry manures and other organic materials having a high organic bound nitrogen (N) content are used for biogas production.

Accordingly, there is provided in one aspect in accordance with the present invention a dual fermentation method for generating biogas from anaerobic fermentation of processed organic material comprising solid and liquid parts, said method comprising the steps of

i) subjecting an organic material comprising one or more sources of nitrogen to a facultative anaerobic fermentation resulting in at least partial hydrolysis of the organic material and at least partial conversion of organic N (nitrogen) present in the organic material to inorganic N, and wherein essentially no biogas is produced;

ii) subjecting the organic material fermented in step i) to lime pressure cooking, wherein the lime-pressure cooking results in a further hydrolysis of said organic material, and wherein said lime pressure cooking step results in the conversion of inorganic N to ammonia fluids which are diverted from the lime pressure cooker and thereby separated from the organic material;

iii) subjecting the organic material fermented in step i) and subjected to lime pressure cooking in step ii) to a strictly anaerobic fermentation resulting in the production of biogas under conditions wherein the pH level of the anaerobic fermenter is kept within a predetermined pH range by contacting or injecting the organic material with fluids comprising CO_2 in amount sufficient to achieve said pH control.

Preferably, the pre-incubated and lime pressure cooked organic material is diverted from said lime pressure cooker to a buffer tank and the pH value of the pre-incubated and lime pressure cooked organic material is lowered by contacting the pre-incubated and lime pressure cooked organic material with with a carbon dioxide (CO_2) containing gas. Optionally, the lowering of the pH value of the pre-incubated and lime pressure cooked organic material in the buffer tank can be assisted by the addition of an acid, such as an organic or inorganic acid, to the organic material.

The diversion to the buffer tank of CO_2 containing gas, or an organic or inorganic acid, controls the pH of the organic material diverted to the buffer tank. The pH of the organic material diverted to the buffer tank is typically above 8.5 when the organic material is initially received from the lime pressure cooker, and it is generally preferred to maintain the pH of the organic material present in the buffer tank within a pH value of from preferably 7.0 to 8.2.

A pH value of from 7.0 to 8.2 is also preferred in the anaerobic digester in which the strictly anaerobic fermentation resulting in the production of biogas is conducted under conditions wherein the pH level is kept within this predetermined pH range in order to reduce the conversion of NH_4^+ to NH_3 as NH_3 is an inhibitor of methanogenic microorganisms.

The formation of NH_3 in significant and undesirable amounts during an anaerobic biogas fermentation will thus result in an inhibition of the methanogenic microorganisms and a reduced biogas yield.

In another aspect of the present invention there is provided a method for generating biogas from an anaerobic fermentation of processed organic material comprising solid and liquid parts, said method comprising the steps of

i) diverting a first organic material comprising one or more sources of nitrogen to a pre-incubation tank and subjecting said organic material to a mineralisation by

chemical and/or biological means, wherein the mineralisation results in the conversion of organic N (nitrogen) present in the organic material to inorganic N;

ii) diverting the pre-incubated first organic material to a lime pressure cooker and subjecting said first pre-incubated organic material to lime pressure cooking, wherein said lime pressure cooking step results in the formation of ammonia fluids which are diverted from the pressure cooker and thereby separated from the organic material present in the lime pressure cooker; and

iii) diverting pre-incubated and lime pressure cooked organic material from said lime pressure cooker to a buffer tank and lowering the pH value of the pre-incubated and lime pressure cooked material in the buffer tank.

The method may comprise the further step of diverting the ammonia fluids formed in the lime pressure cooker to an absorption unit, and absorbing and condensing the ammonia fluids diverted to the absorption unit from the lime pressure cooker.

Also, in a still further step, the pre-incubated and lime pressure cooked organic material may be mixed in the buffer tank with a further organic material; wherein the further organic material preferably has not been subjected to lime pressure cooking prior to the mixing with the lime pressure cooked organic material.

The pH value of the pre-incubated and lime pressure cooked organic material which is diverted from the lime pressure cooker to the buffer tank is lowered in the buffer tank by contacting the pre-incubated and lime pressure cooked organic material in the buffer tank with a carbon dioxide (CO_2) containing gas. The lowering of the pH value of the organic material in the buffer tank may optionally be assisted by the addition of an acid to the organic material.

The carbon dioxide (CO_2) containing gas diverted to the organic material in the buffer tank can be biogas diverted to the buffer tank from an anaerobic fermenter which is receiving as input biomass material the organic material present in the buffer tank.

The diversion to the buffer tank of CO_2 containing gas controls the pH of the organic material present in the buffer tank. The pH of the organic material diverted to the buffer tank is typically above 8.5 when initially received from the lime pressure cooker, and it is generally preferred to

maintain the pH of the organic material present in the buffer tank within a pH value of from preferably 7.0 to 8.2.

A pH value of from 7.0 to 8.2 is also preferred in the anaerobic digester in which the strictly anaerobic fermentation resulting in the production of biogas is conducted under conditions wherein the pH level is kept within this predetermined pH range. This pH interval is preferred in order to reduce the conversion of NH_4^+ to NH_3 as NH_3 is an inhibitor of methanogenic microorganisms.

Following the adjustment of the pH of the organic material to a pH value of from preferably pH = 7.0 til pH = 8.2, the optionally mixed, organic material(s) are diverted from the buffer tank to at least one, anaerobic biogas fermenter suitable for conducting an anaerobic, bacterial digestion of the organic material, wherein the anaerobic, bacterial digestion results in the generation of biogas by fermenting, under anaerobic fermentation conditions, the organic material diverted to the anaerobic biogas fermenter from the buffer tank, and collecting the biogas resulting from said anaerobic fermentation of said organic material.

The pH value of the organic material diverted to the anaerobic biogas fermenter can optionally be maintained in the anaerobic biogas fermenter within a predetermined pH-range by also contacting the organic material present in the anaerobic biogas fermenter with re-circulated biogas, or a biogas diverted to the anaerobic biogas fermenter from an external source, wherein said contacting of said biogas with said organic material results in said pH value being maintained within a predetermined pH-range, preferably a pH-range of from pH = 7.0 to pH = 8.2.

Accordingly, it is possible to adjust and control the pH value of the organic material present in the buffer tank by diverting a CO_2 containing gas to the organic material present in the buffer tank. If it is deemed relevant or desirable, it is furthermore possible to further adjust and control the pH value of the organic material when the organic material has been diverted from the buffer tank and entered into the anaerobic digester. In the latter case the pH is adjusted and controlled by diversion of CO_2 containing gas to the anaerobic fermenter containing the organic material.

According to yet another aspect of the invention there is provided a method for generating biogas from an anaerobic fermentation of processed organic material, including solid and liquid parts, is disclosed. The method includes i) diverting a first organic material comprising one or more sources of nitrogen to a lime pressure cooker; ii) subjecting said first organic material to a lime pressure cooking step resulting in at least partly hydrolysing said first organic material

comprising one or more sources of nitrogen, wherein said lime pressure cooking step results in the formation of ammonia fluids; iii) diverting said ammonia fluids formed in the lime pressure cooker to an absorption unit; iv) absorbing and condensing ammonia fluids diverted to the absorption unit from the lime pressure cooker; v) diverting lime pressure cooked organic material from said lime pressure cooker to a buffer tank; vi) mixing lime pressure cooked organic material with a further organic material in the buffer tank; vii) contacting the mixed, organic materials with a CO_2 containing gas, such as e.g. biogas, in order to control the pH of the mixed organic materials prior to anaerobic digestions and biogas production within a range of from preferably pH = 7.0 to pH = 8.2; viii) diverting the mixed, organic materials to one or more fermenters; ix) fermenting, under anaerobic fermentation conditions said mixed, organic materials in said one or more fermenters, x) generating biogas from said anaerobic fermentation of said mixed, organic materials, wherein the pH of said mixed, organic materials is controlled by contacting the mixed, organic materials with a CO_2 containing gas, such as e.g. biogas, in order to preferably maintain the pH of the mixed, organic materials present in the one or more fermenters within a range of from preferably pH = 7.0 to pH = 8.2; xi) diverting said mixed, organic materials from said one or more fermenters to a separation unit; xii) separating said mixed, organic materials into a solid organic material fraction and a liquid fraction comprising one or more sources of nitrogen; and xiii) diverting said liquid fraction comprising one or more sources of nitrogen to the lime pressure cooker, xiv) optionally mixing said liquid fraction comprising one or more sources of nitrogen with a first organic material and/or a further organic material comprising one or more sources of nitrogen, and xv) stripping ammonia from said liquid fraction, or from the mixture of liquid fraction and said first and/or further organic material obtained in step xiv).

In a still further aspect of the present invention there is provided a method for generating biogas from an anaerobic fermentation of processed organic material comprising solid and liquid parts, said method comprising the steps of

i) diverting a first organic material comprising one or more sources of nitrogen to a pre-incubation tank and subjecting said organic material to a mineralisation by chemical and/or biological means, wherein the mineralisation results in the conversion of organic N (nitrogen) present in the organic material to inorganic N;

ii) diverting the pre-incubated first organic material to a lime pressure cooker and subjecting said first pre-incubated organic material to lime pressure cooking,

wherein said lime pressure cooking step results in the formation of ammonia fluids;

iii) diverting pre-incubated and lime pressure cooked organic material from said lime pressure cooker to a buffer tank and lowering the pH value of the pre-incubated and lime pressure cooked material by contacting the pre-incubated and lime pressure cooked organic material with a carbon dioxide (CO_2) containing gas.

In an even further aspect of the present invention there is provided a method for generating biogas from an anaerobic fermentation of processed organic material comprising solid and liquid parts, said method comprising the steps of

i) diverting a first organic material comprising one or more sources of nitrogen to a pre-incubation tank and subjecting said organic material to a mineralisation by chemical and/or biological means, wherein the mineralisation results in the conversion of organic N (nitrogen) present in the organic material to inorganic N;

ii) diverting the pre-incubated first organic material to a lime pressure cooker and subjecting said first pre-incubated organic material to lime pressure cooking, wherein said lime pressure cooking step results in the formation of ammonia fluids;

iii) diverting pre-incubated and lime pressure cooked organic material from said lime pressure cooker to a buffer tank and lowering the pH value of the pre-incubated and lime pressure cooked material in the buffer tank.

Accordingly, as will be clear from the above, the present invention generally provides improved methods for processing organic biomass materials, converting organic nitrogen (N) fractions, including protein N and uric acid, into inorganic nitrogen (N) fractions by mineralization, removing by stripping or extraction inorganic nitrogen (N) by subjecting the organic biomass materials to a temperature and pH which allow gaseous ammonia to be stripped or extracted, thereby reducing the contents of inorganic nitrogen (N) fractions, including ammonia, which would otherwise cause an inhibitory effect in a subsequent, anaerobic biogas fermentation, and, as a result of reducing said inorganic nitrogen (N) fractions from the organic biomass to be subjected to anaerobic biogas fermentation, increasing the production of biogas when

performing the anaerobic biogas fermentation on the organic biomass having reduced contents of otherwise inhibitory, or potentially inhibitory, organic and/or inorganic nitrogen (N) fractions.

Under various practical circumstances the methods of the present invention can achieve one or more of the below-cited technical effects in respect of the processing of an organic biomass material prior to subjecting the biomass material to an anaerobic biogas fermentation.

It is possible, in accordance with the methods of the present invention, to convert essentially all of the uric acid present in the layer manure biomass to total ammonia N (TAN) during a biological, facultative anaerobic pre-treatment step as disclosed herein elsewhere in more detail.

It is also possible, in accordance with the methods of the present invention, to convert at least approximately 50%, such as for example at least approximately 55%, for example at least approximately 60%, such as for example at least approximately 65%, for example at least approximately 70%, such as for example at least approximately 75%, for example at least approximately 80%, of the organic N to inorganic N during the biological pre-treatment step as disclosed herein elsewhere in more detail.

It is also possible, in accordance with the methods of the present invention, to increase at least by a factor of approximately 3, such as at least by a factor of approximately 4, for example at least by a factor of approximately 5, as a result of the afore-mentioned organic material N conversions, the amount of total ammonia N (TAN) available for ammonia stripping during the (NiX) nitrogen extracting, thermo-chemical lime pressure cooking step as disclosed herein elsewhere in more detail.

It is also possible, in accordance with the methods of the present invention, to strip at least approximately 65%, such as at least approximately 70%, for example at least approximately 75%, such as at least approximately 80%, for example at least approximately 85%, of the total ammonia N TAN during the (NiX) nitrogen extracting, thermo-chemical lime pressure cooking step.

It is also possible, in accordance with the methods of the present invention, to reduce by at least approximately 50%, such as by at least approximately 55%, for example by at least approximately 60%, such as by at least approximately 65%, for example by at least approximately 70%; such as by at least approximately 75%, for example by at least approximately 80% - as a result of the stripping of total ammonia N (TAN) - the overall TKN

value for the layer manure biomass in question, thereby significantly alleviating any inhibitory effect on the biogas formation exerted by the formation of ammonia in undesirable amounts during a subsequent biogas fermentation, i.e. amounts of ammonia which would have developed during the subsequent, anaerobic biogas fermentation in case the TKN value of the layer manure biomass had not been reduced as described herein elsewhere in more detail,

It is also possible, in accordance with the methods of the present invention, to convert the ratio of organic N : inorganic N from a ratio of more than 3 : 1 (gram organic N/kg TS : gram inorganic N/kg TS) prior to performing the biological, facultative anaerobic pre-treatment step of the methods of the present invention, to a ratio of at the most 1 : 1 (gram organic N/kg TS : gram inorganic N/kg TS), or less, such as a ratio of at the most 4 : 5 (gram organic N/kg TS : gram inorganic N/kg TS), for example a ratio of at the most 2 : 3 (gram organic N/kg TS : gram inorganic N/kg TS), such as a ratio of at the most 3 : 5 (gram organic N/kg TS : gram inorganic N/kg TS), for example a ratio of at the most 4 : 7 (gram organic N/kg TS : gram inorganic N/kg TS), prior to performing an anaerobic biogas fermentation, depending on the amount of organic N converted during the biological pre-treatment step and depending on the amount of TAN stripped during the (NiX) thermo-chemical lime pressure cooking step.

By diverting e.g. a CO_2 containing gas to the organic biomass material present in the buffer tank, or in the anaerobic fermenter, it is possible to control the pH of the organic biomass material and to preferably maintain said pH of said organic biomass material within a range of from pH = 7.0 to preferably less than pH = 8.2, thereby further stabilizing the conditions needed for optimizing the production of biogas during the anaerobic digestion in the anaerobic fermenter.

According to a still further aspect of the invention there is provided a plant for generating biogas from an anaerobic fermentation of processed organic material, including solid and liquid parts, is disclosed. The plant includes i) a lime pressure cooker for hydrolysing a first organic material comprising one or more sources of nitrogen; ii) an absorption unit for absorbing and condensing ammonia fluids diverted to the absorption unit from the lime pressure cooker when said first organic material is subjected to lime pressure cooking; iii) a buffer tank for mixing lime pressure cooked organic material with a further organic material prior to diverting the mixed, organic materials to one or more fermenters; iv) one or more fermenters for anaerobically fermenting said organic materials diverted to said one or more fermenters from said buffer tank, wherein said fermentation results in the generation of biogas; v) a separation unit for separating organic materials diverted to the separation unit from said one or more fermenters, wherein said separation of said organic materials results in the generation of a solid organic

material fraction and a liquid fraction comprising one or more sources of nitrogen; vi) a means for diverting said liquid fraction comprising one or more sources of nitrogen to the lime pressure cooker, wherein said liquid fraction is mixed in the lime pressure cooker with first organic material comprising one or more sources of nitrogen; and vii) a means for diverting a CO_2 containing gas, such as e.g. biogas, into the organic biomaterials present in the one or more fermenters in order to control the pH of the biomaterials during biogas production.

Brief Description of the Figures

The embodiments of the invention, together with its advantages, may be best understood from the following detailed description taken in conjunction with the accompanying figures.

Figures 1 to 24 illustrate various alternative embodiments of the present invention in which organic biomass and re-circulated liquid from an anaerobic digester (biogas fermenter(s)) are diverted to a lime pressure cooking step (NiX treatment) and lime (CaO / $Ca(OH)_2$ is added to create - at a temperature of more than 100°C and a pressure of more than 1 bar - a pH suitable for converting ammonium N (NH_4^+) to gaseous ammonia (NH_3) - which can be stripped, collected e.g. in an ammonia scrubber, and converted into ammonium sulphate - which can subsequently be used as a fertilizer.

Following ammonia stripping in the lime pressure cooker, the lime pressure cooked biomass is diverted to a buffer tank - prior to being diverted into one, or a series of at least two, connected anaerobic digesters for the production of biogas.

Additional biomass may be mixed with the lime pressure cooked biomass in the buffer tank.

A conversion of organic nitrogen to ammonium N (NH_4^+) may take place in the buffer tank – and the generated ammonium N (NH_4^+) may subsequently be converted to gaseous ammonia (NH_3) under suitable conditions.

Following anaerobic digestion and biogas production, a separation of fiber (i.e. solid fraction, “spent biomass”, also known as the digestate) and liquid phase takes place and the liquid fraction can be diverted (i.e. re-circulated) back to the lime pressure cooker.

In order to control the pH value of the organic biomass during the processing thereof, a CO_2 containing gas, such as e.g. biogas or flue gas, is added or injected on gaseous form to the organic biomass present in the buffer tank and/or in the anaerobic digester.

Figures 1 to 6 illustrate a plant and a process as described herein above in which a pre-incubation step has been inserted prior to Nix treatment / lime pressure cooking. Under suitable conditions, the biomass is being converted into more basic constituents, i.e. fx peptides, saccharides and fatty acids, chemically and/or biologically under anaerobic and/or aerobic conditions, and organic N - i.e. organic nitrogen bound in and forming part of the biomass, is subsequently mineralised and converted into ammonium N (NH_4^+) – which in turn, again under suitable conditions, can be converted into gaseous ammonia (NH_3). Depending on the retention time and reactions conditions in the pre-incubation tank and in the buffer tank, respectively, these two supplementary pre-treatment steps may aid significantly in the conversion / mineralisation of organic nitrogen to inorganic nitrogen – i.e. what is also termed N-mineralization.

In the embodiments illustrated in Figures 1 to 6, ammonia is being stripped from the lime pressure cooker only. In Fig. 1, a CO_2 containing gas is being added or injected into the organic biomaterial present in the buffer tank. In Fig. 2, a CO_2 containing gas, preferably biogas, is being added or injected into the organic biomaterial present in the anaerobic digester. In Fig. 3, a CO_2 containing gas is being added or injected into the organic biomaterial present in the buffer tank as well as into the anaerobic digester. In all cases, the addition or injection of the CO_2 containing gas is aimed at controlling the pH value of the organic biomass as explained herein below in the detailed description as well as in the examples. Figures 4 to 6 illustrate the embodiments illustrated in Figures 1 to 3 with the addition of further organic biomass to the anaerobic digester. For all of the illustrated embodiments it is possible to divert liquid fraction from the anaerobic digester to the buffer tank (not illustrated).

Figures 7 to 12 illustrate embodiments in which an addition of lime to the pre-incubation tank is performed in order to increase the conversion of organic N to ammonium N (NH_4^+) - and to shift the equilibrium between ammonium N (NH_4^+) and gaseous ammonia (NH_3) in the direction of gaseous ammonia (NH_3) - with a view to stripping gaseous ammonia (NH_3) also during the pre-incubation phase. Additional lime may be added during the lime pressure cooking step (not shown).

Accordingly, in the embodiments illustrated in Figures 7 to 12, ammonia is being stripped not only from the lime pressure cooker, but also from the pre-incubation tank. In Fig. 7, a CO_2

containing gas is being added or injected into the organic biomaterial present in the buffer tank. In Fig. 8, a CO_2 containing gas, preferably biogas, is being added or injected into the organic biomaterial present in the anaerobic digester. In Fig. 9, a CO_2 containing gas is being added or injected into the organic biomaterial present in the buffer tank as well as into the anaerobic digester. In all cases, the addition or injection of the CO_2 containing gas is aimed at controlling the pH value of the organic biomass as explained herein below in the detailed description as well as in the examples. Figures 10 to 12 illustrate the embodiments illustrated in Figures 7 to 9 with the addition of further organic biomass to the anaerobic digester. For all of the illustrated embodiments it is possible to divert liquid fraction from the anaerobic digester to the buffer tank (not illustrated).

Figures 13 to 18 illustrate embodiments in which ammonia is stripped from the buffer tank following NiX treatment (i.e. lime pressure cooking) - in addition to being stripped during the lime pressure cooking step (i.e. nitrogen extraction - NiX treatment).

Accordingly, in the embodiments illustrated in Figures 13 to 18, ammonia is being stripped not only from the lime pressure cooker, but also from the buffer tank. In Fig. 13, a CO_2 containing gas is being added or injected into the organic biomaterial present in the buffer tank. In Fig. 14, a CO_2 containing gas, preferably biogas, is being added or injected into the organic biomaterial present in the anaerobic digester. In Fig. 15, a CO_2 containing gas is being added or injected into the organic biomaterial present in the buffer tank as well as into the anaerobic digester. In all cases, the addition or injection of the CO_2 containing gas is aimed at controlling the pH value of the organic biomass as explained herein below in the detailed description as well as in the examples. Figures 16 to 18 illustrate the embodiments illustrated in Figures 13 to 15 with the addition of further organic biomass to the anaerobic digester. For all of the illustrated embodiments it is possible to divert liquid fraction from the anaerobic digester to the buffer tank (not illustrated).

Figures 19 to 24 illustrate embodiments in which gaseous ammonia (NH_3) is stripped from each and all of the pre-incubation tank, the lime pressure cooker (NiX treatment) and the buffer tank. It is illustrated that lime (CaO) is added to the pre-incubation tank, but additional lime may be added to the lime pressure cooker, if needed. The operational conditions as well as the chemical reaction conditions are different for the pre-incubation tank and for the lime pressure cooker as described herein below in more detail.

Accordingly, in the embodiments illustrated in Figures 19 to 24, ammonia is being stripped not only from the lime pressure cooker, but also from the pre-incubation tank prior to lime pressure

cooking, and from the buffer tank following the lime pressure cooking step. In Fig. 19, a CO_2 containing gas is being added or injected into the organic biomaterial present in the buffer tank in order to control the pH. In Fig. 20, a CO_2 containing gas, preferably biogas, is being added or injected into the organic biomaterial present in the anaerobic digester. In Fig. 21, a CO_2 containing gas is being added or injected into the organic biomaterial present in the buffer tank as well as into the anaerobic digester. In all cases, the addition or injection of the CO_2 containing gas is aimed at controlling the pH value of the organic biomass as explained herein below in the detailed description as well as in the examples. Figures 22 to 24 illustrate the embodiments illustrated in Figures 19 to 21 with the addition of further organic biomass to the anaerobic digester. For all of the illustrated embodiments it is possible to divert liquid fraction from the anaerobic digester to the buffer tank (not illustrated).

Figure 25 illustrates a time response curve for pH neutralisation of a lime pressure cooked (NiX treated) organic biomass material.

Figure 26 illustrates expected methane production in a two-stage CSTR with a thermophilic primary digester and a mesophilic secondary digester, both with 15 days retention time in accordance with the Example 3. Bt = methane yield after time t. Error bars are produced from 95% confidence intervals (see Fig. 28 and Table 7);

Figure 27 illustrates Retford hen litter in accordance with the Example 3;

Figure 28 illustrates specific methane yield from batch bottles with Hen litter in accordance with the Example 3. Errorbars equal 1x the standard deviation. B(t) – measured methane yield.

Figure 29 illustrates specific methane yield per kg chicken litter VS added (5 days rolling average) in accordance with the Example 4;

Figure 30 illustrates TAN and NH3 concentrations in the digester in accordance with the Example 4;

Figure 31 illustrates pH in the digester in accordance with the Example 4;

Figure 32 illustrates VFA in the digester in accordance with the Example 4;

Figure 33 illustrates Blow-up of C4-C5 VFA in the digester in accordance with the Example 4;

Figure 34 illustrates TS in digester and recycled liquid in accordance with the Example 4;

Figure 35 illustrates specific methane yield per kg chicken litter VS added (5 days rolling average) in accordance with the Example 4;

Figure 36 illustrates TAN and NH_3 concentrations in the digester in accordance with the Example 4.

Figure 37 illustrates pH in the digester in accordance with the Example 4;

Figure 38 illustrates VFA in the digester in accordance with the Example 4;

Figure 39 illustrates Blow-up of C4-C5 VFA in digester in accordance with the Example 4;

Figure 40 illustrates TS in digester and recycled liquid in accordance with the Example 4;

Figure 41 illustrates specific methane yield per kg chicken litter VS added (5 days rolling average) in accordance with the Example 4;

Figure 42 illustrates TAN and NH_3 concentrations in the digester in accordance with the Example 4;

Figure 43 illustrates pH in the digester in accordance with the Example 4;

Figure 441 VFA in the digester in accordance with the Example 4;

Figure 45 illustrates Blow-up of C4-C5 VFA in digester in accordance with the Example 4;

Figure 46 illustrates TS in the digester and recycled liquid in accordance with the Example 4;

Figure 47 illustrates specific methane yield per kg chicken litter VS added (5 days rolling average) in accordance with the Example 4;

Figure 48 illustrates TAN and NH3 concentrations in the digester in accordance with the Example 4;

Figure 49 illustrates pH in the digester in accordance with the Example 4;

Figure 50 illustrates VFA in digester in accordance with the Example 4;

Figure 51 illustrates Blow-up of C4-C5 VFA in digester in accordance with the Example 4;

Figure 52 illustrates TS in digester and recycled liquid in accordance with the Example 4;

Figure 53 illustrates TAN and NH_3 concentrations in the digester in accordance with the Example 4;

Figure 54 illustrates pH in the digester in accordance with the Example 4;

Figure 55 illustrates TAN and NH_3 concentrations in the digester in accordance with the Example 4;

Figure 56 illustrates pH in the digester in accordance with the Example 4;

Figure 57 illustrates specific methane yield per kg chicken litter VS added (7 day rolling average) in accordance with the Example 4;

Figure 58 illustrates VFA in digester in accordance with the Example 4;

Figure 59 illustrates TS in digester and recycled liquid in accordance with the Example 4;

Figure 60 CH_4% in the biogas in accordance with the Example 4;

Figure 61 illustrates expected methane yields of broiler litter in a CSTR setup in accordance to the Example 5. 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 62 illustrates accumulated methane yield from BMP setup 1 with untreated, mineralised and NiX treated chicken litter in accordance with the Example 5. Errorbars represent 95% confidence intervals;

Figure 63 illustrates accumulated methane yield from BMP setup 2 with untreated, mineralised, NiX treated and pH adjusted chicken litter in accordance with the Example 5. Errorbars represent 95% confidence intervals;

Figure 64 illustrates precipitated material during pH neutralisation of organic biomass using $CO_{2(g)}$

Detailed description of the Invention

There are four key biological and chemical stages of an anaerobic fermentation: Hydrolysis; acidogenesis; acetogenesis; and methanogenesis. In order for bacteria under anaerobic conditions to exploit the energy potential of the organic materials used as substrates, macromolecules present in the organic materials must initially be broken down into their smaller constituent parts - unless this has already occurred at an earlier processing step, such as e.g. lime pressure cooking.

Lime pressure cooking is an example of a pre-treatment processing step resulting in nitrogen extraction - a technical term often abbreviated "NiX" (cf. Figures 1 to 24). Lime pressure cooking is in principle conducted at temperatures above 100°C and at a pressure of above 1 bar. Lime pressure cooking results in the conversion of inorganic ammonia N (MH_4^+) to gaseous ammonia (NH_3) as illustrated in Figures 1 to 24. The term pre-treatment signifies that this processing step occurs prior to the step of anaerobic digestion and the production of biogas.

When a biomaterial is subjected to lime pressure cooking priot to anaerobic digestion and biogas production in accordance with the principles of the present invention, the pH of the treated biomaterial is increased by adding (burnt) lime (CaO) to the biomaterial.

Addition of lime facilitates a reduction in the TAN pool by facilitating a removal of ammonia fluids. This is a necessary step in order to obtain high ammonia removal efficiencies (see Equation 3 and Equation 4).

$$CaO_{(s)} + H_2O_{(l)} \leftrightarrow Ca^{2+}_{(aq)} + 2OH^-_{(aq)}$$

Equation 1

$$2NH_{4\,(aq)}^{+} + 2OH_{(aq)}^{-} \leftrightarrow 2NH_{3(g)} + 2H_2O_{(l)}$$

Equation 2

One disadvantage associated with using this method is the low solubility of $CaO_{(s)}$. The low solubility may under some practical conditions result in substantial amounts of residual $CaO_{(s)}$ after the NiX treatment / lime pressure cooking step. The surplus of alkaline constituents will subsequently be dissolved in the anaerobic digester – and this leads to an undesirably increase in the pH value of the biomaterial to be subjected to anaerobic digestion.

The equilibrium between ammonia ($NH_{3(g)}$) and ammonium (NH_4^+) is pH dependent. More ammonia ($NH_{3(g)}$) will be present with higher pH levels. As ammonia exerts an inhibitory effect on the microorganisms responsible for fermenting the biomaterial and producing the biogas, ammonia can be regarded as an undesirable part of the TAN (total ammonium Nitrogen) pool, and any residual alkaline constituents present in the anaerobic digester is likely to counteract the benefits of the TAN removal obtained by performing a NiX treatment / lime pressure cooking.

Another disadvantage associated with using lime is the potential build-up of calcium ions (Ca^{2+}) – a build-up which may be detrimental to the stability of the anaerobic digester. It is well known that anaerobic digestion is sensitive to the osmotic potential. However, very little has been published in the literature with respect to the effect of the concentration of Ca^{2+} on the stability of an anaerobic digester.

The present invention solves the above-cited disadvantages by lowering the pH of the biomaterial present in a buffer tank prior to anaerobic digestion, or by lowering the pH of the biomaterial present in the anaerobic digester itself.

The solution involves one or more step(s) associated with injecting a carbon dioxide containing gas ($CO_{2(g)}$) - such as e.g. biogas, which contain carbon dioxide in amounts relevant for the purpose of and practical solution provided by the present invention - through the NiX treated / lime pressure cooked biomaterial either when the NiX treated / lime pressure cooked biomaterial is present in a buffer tank following lime pressure cooking, and/or when the NiX treated / lime pressure cooked biomaterial has subsequently been diverted to the anaerobic digester.

It is well known that $CO_{2(g)}$ can be converted to carbonic acid ($H_2CO_{3(aq)}$) if it reacts with water, and this will lead to a neutralisation of exogenous base.

The present invention demonstrates that it is possible to lower the pH of a biomaterial by injecting $CO_{2(g)}$ containing fluids into the biomaterial following NiX treatment / lime pressure cooking, and that calcium can be precipitated as calcium carbonate ($CaCO_{3(s)}$) during the neutralisation reaction.

One conceivable driving force for the reaction is likely the precipitation of $CaCO_{3(s)}$, which continuously removes the produced carbonate. Importantly, this mechanism may also explain why the pH of a biomaterial can be lowered to a lower level than the pH value of the original substrate prior to injection in this substrate of $CO_{2(g)}$ containing fluids.

As part of the hydroxyl ions (OH^-) have already been neutralised during TAN removal, the residual Ca^{2+} ions have a potential - through the formation of $H_2CO_{3(aq)}$ - to drive the pH level below that of the original pH level of the biomaterial.

The formation of the ammonia fraction of the TAN pool is pH dependent and one advantage of acidifying the NiX treated / lime pressure cooked biomaterial is that this in itself creates the possibility of reducing the amount of biomaterial that will have to be NiX treated / lime pressure cooked.

The possibility of reducing the amount of biomaterial that will have to be NiX treated / lime pressure cooked in turn saves energy and reduces the cost of performing a NiX treatment / lime pressure cooking step.

The acidification of the biomaterial by injection of $CO_{2(g)}$ containing fluids can be performed in a buffer tank following the lime pressure cooking step. This is illustrated e.g. in the embodiments illustrated in the enclosed figures 1, 3, 4, 6, 7, 9, 10, 12, 13, 15, 16, 18, 19, 21, 22, and 24.

Alternatively, as well as by means of an additional $CO_{2(g)}$ fluid injection step, $CO_{2(g)}$ containing fluids can be injected into a biomaterial present in an anaerobic digester - to which the optionally $CO_{2(g)}$ treated biomaterial in the buffer tank can be diverted.

The acidification of the biomaterial by injection of $CO_{2(g)}$ containing fluids can be performed directly in an anaerobic digester following diversion of biomaterial from the buffer tank to the anaerobic

digester. This is illustrated e.g. in the embodiments illustrated in the enclosed figures 2, 3, 5, 6, 8, 9, 11, 12, 14, 15, 17, 18, 20, 21, 23 and 24.

Diversion of $CO_{2(g)}$ containing fluids, including diversion of biogas, to one or more biomaterials in both in the buffer tank as well as in the anaerobic digester is illustrated in figures 3, 6, 9, 12, 15, 18, 21 and 24.

Accordingly, in accordance with the present invention, the pH value of the organic biomaterial diverted to the anaerobic biogas fermenter can be maintained within a predetermined pH-range by contacting the organic material present in the anaerobic biogas fermenter e.g. with re-circulated biogas, or a biogas diverted to the anaerobic biogas fermenter from an external source, wherein said contacting results in said pH value of the biomaterial being maintained within a predetermined pH-range.

It is also possible to obtain an additional contribution to the lowering of the pH value of the biomaterial present in the buffer tank. At least part of a liquid organic material fraction comprising one or more sources of nitrogen selected from organic nitrogen sources and inorganic nitrogen sources and obtained by separation of the liquid fraction from a solid fraction following anaerobic digestion and biogas production can be diverted to the buffer tank, wherein said diversion of said liquid organic material fraction results in, or contributes to, lowering the pH-value of the pre-incubated and lime pressure cooked organic material present in the buffer tank.

The pH value of the pre-incubated material subjected to lime pressure cooking in the lime pressure cooker is preferably kept, during the lime pressure cooking treatment, within a pH range of from pH = 7.5 to pH = 12.0; for example from pH = 7.5 to pH = 11.5, such as from pH = 7.5 to pH = 11.0; for example from pH = 7.5 to pH = 10.5; such as from pH = 7.5 to pH = 10.0; for example from pH = 7.5 to pH = 9.5; such as from pH = 7.5 to pH = 9.0.

The pH value of the pre-incubated material subjected to lime pressure cooking in the lime pressure cooker decreases over time due to the stripping of gaseous ammonia. The initial pH value of the pre-incubated material to which lime has been added, prior to the pressure cooking, is preferably within a range of from more than pH = 9.5 to pH = 12.0; and the terminal pH value of the pre-incubated and lime-pressure cooked material, i.e. after completion of the lime pressure cooking step, is preferably within a range of from pH = 7.5 to pH = 9.5.

The pH value in the buffer tank of the pre-incubated and lime pressure cooked organic material is preferably kept within a pH range of from pH = 7.0 to pH = 9.5; for example from pH = 7.0 to pH =

9.2, such as from pH = 7.0 to pH = 9.0; for example from pH = 7.0 to pH = 8.7; such as from pH = 7.0 to pH = 8.5; for example from pH = 7.0 to pH = 8.2; such as from pH = 7.0 to pH = 8.0; for example a pH of around 7.5.

The pH value in the buffer tank of the pre-incubated and lime pressure cooked organic material will be lower than the pH value of the pre-incubated and lime pressure cooked organic material present in the lime pressure cooker because of the addition or diversion to the buffer tank of pH lowering means.

In principle, the lowering of the pH value of the lime pressure cooked organic material present in the buffer tank can be obtained i) by contacting said organic material in the buffer tank with a CO_2 containing gas, such as biogas, and/or ii) by contacting said organic material in the buffer tank with an acid selected from an organic acid and an inorganic acid, and/or iii) by diverting, following anaerobic digestion and separation of the fermented, organic material, the separated liquid organic material fraction comprising one or more sources of nitrogen selected from organic nitrogen sources and inorganic nitrogen sources to the buffer tank

The predetermined pH value of the organic material present in the anaerobic digester is preferably kept within a pH range of from pH = 7.0 to pH = 8.5; for example from pH = 7.0 to pH = 8.2, such as from pH = 7.0 to pH = 8.0; for example from pH = 7.0 to pH = 7.8; such as from pH = 7.0 to pH = 7.5; such as from pH = 7.2 to pH = 8.5; for example from pH = 7.2 to pH = 8.2, such as from pH = 7.2 to pH = 8.0; for example from pH = 7.2 to pH = 7.8; such as from pH = 7.2 to pH = 7.5, for example from pH = 7.4 to pH = 8.5; for example from pH = 7.4 to pH = 8.2, such as from pH = 7.4 to pH = 8.0; for example from pH = 7.4 to pH = 7.8.

An additional pre-treatment processing step, in addition to NiX (nitrogen extraction) treatment / lime pressure cooking, which occurs prior to the step of lime pressure cooking, is that of pre-incubation of the biomass to be lime pressure cooked and later subjected to anaerobic digestion.

This additional pre-incubation step also serves to improve biogas production by converting a biomass into smaller constituent parts by chemical hydrolysis, or by biological conversion, using one or more microbial populations and/or one or more enzymes.

The initial process of breaking down macromolecular structures of a substrate in an anaerobic fermentation involves a hydrolysis of macromolecular structures, such as proteins, carbohydrates and organic acids. The constituent parts, or monomers, such as amino acid residues, sugars and fatty acids can be readily metabolized by microbial organisms. Accordingly, hydrolysis of

macromolecular components of organic materials represents an initial step in an anaerobic fermentation.

Anaerobic fermentations are sensitive to high levels of ammonia as the ammonia inhibits the bacteria which are responsible for the methanogenesis. Hence, when the bacteria are inhibited by high levels of ammonia, reduced amounts of biogas are being produced.

In order to prevent ammonia inhibition, or to reduce the problem of ammonia inhibition during an anaerobic fermentation resulting in a reduced production of biogas, it is necessary to remove significant parts of the nitrogen which is present in an organic biomass.

The pre-incubation steps of the methods of the present invention are aimed at increasing the removal of nitrogen sources from an organic biomass. In combination with the subsequent lime pressure cooking step, during which ammonia is stripped, the pre-incubation step serves to effectively prevent ammonia inhibition during anaerobic fermentation and biogas production.

Nitrogen can be present in an organic biomass either as organic nitrogen – fx nitrogen present in proteins and organic acids – or as inorganic nitrogen – in the form of ammonium. In order to strip gaseous ammonia from the lime pressure cooker, the organic bound nitrogen will have to initially be converted into inorganic ammonium, which is then stripped in the form gaseous ammonia. This is performed under suitable conditions – primarily involving a high pH and an increased temperature.

However, it has been observed that organic nitrogen cannot - or only to a very limited extent - be converted into inorganic nitrogen during a lime pressure cooking step. Hence, the ammonium available for stripping in the lime pressure cooker is determined by the amount of inorganic nitrogen which is entered into the lime pressure cooker for ammonia stripping. Essentially no conversion of organic nitrogen into inorganic nitrogen is expected to take place during lime pressure cooking.

This poses a significant challenge to anaerobic biogas fermentations - as the organic material will contain significant amounts of organic nitrogen - which is not converted into removable ammonia during lime pressure cooking.

In order to increase the amount of inorganic nitrogen available for ammonia stripping in the lime pressure cooking step, a further pre-treatment step in the form of a pre-incubation of an organic biomass is introduced. The pre-incubation step comprises one or both of a pre-

fermentation step and/or a chemical hydrolysis and N mineralisation step. Pre-fermentation can result in a hydrolysis and/or further break-down of e.g. proteins, carbohydrates and other macromolecules present in an organic biomass. Hence, hydrolysis of macromolecules can also be obtained by microbial means – and not exclusively by chemical means. N mineralisation involves the conversion of organic N into inorganic N.

According to one presently preferred hypothesis, the pre-incubation step comprises a microbial fermentation resulting in the decomposition of organic macromolecules present in the organic material which is to be subsequently subjected to anaerobic fermentation and biogas fermentation.

The microbial fermentation and/or hydrolysis of macromolecules present in an organic material which takes place during the pre-incubation step will thus contribute to an increased N-mineralization process during the pre-incubation step(s).

Accordingly, the conversion of organic N to inorganic N which takes place at the pre-incubation step is in one embodiment of the present invention at least facilitated by biological and enzymatic processes catalyzed by microbial organisms present in the biomass comprising the organic bound N.

Without being bound by theory, and according to one presently preferred hypothesis, enzymes synthesized by microbial organisms residing in the organic material to be processed are largely responsible for the conversion of organic N to inorganic N during pre-incubation - and such enzymes will not be allowed to exert their action during a lime pressure cooking step - which is a thermo-chemical process. Hence, it is likely that this represents one explanation as to why organic bound N can be converted into inorganic N when being subjected to enzymatic action during pre-incubation, whereas no, or only a very modest part, of the organic bound N is converted to inorganic N during the thermo-chemical lime pressure cooking step under conditions, when no enzyme is active.

It has been observed that the microbial organisms capable of contributing to the conversion of organic N to inorganic N during the pre-incubation step are apparently not involved in biogas production - as essentially no methanogenesis takes place during pre-incubation.

Accordingly, in view of the above observation, the methods of the present invention can be characterized as a two-step fermentation method in which the individual steps are separated

by a thermo-chemical processing step - i.e. lime pressure cooking - performed at an elevated temperature and pressure, and under alkaline pH conditions.

The first fermentation step - preceding the thermo-chemical processing step - is a facultative anaerobic fermentation reaction during which, in one embodiment of the present invention, essentially no biogas is produced - as the organic material can be expected to undergo initial fermentation stages during the first fermentation step, but not, or only to a limited extent, methanogenesis. Methanogenesis constitutes one of the latter stages of an anaerobic fermentation - i.e. a stage which is reached only after prior stages, such as e.g. acidogenesis and acetogenesis.

The second fermentation step, which takes place after the thermo-chemical processing step - is a strictly anaerobic methanogenesis. Accordingly, the second fermentation step is aimed at producing biogas by using the pre-fermented and thermo-chemically treated organic material as a substrate.

Accordingly, there is also provided in accordance with the present invention a dual fermentation method for generating biogas from anaerobic fermentation of processed organic material comprising solid and liquid parts, said method comprising the steps of

i) subjecting an organic material comprising one or more sources of nitrogen to a facultative anaerobic fermentation resulting in at least partial hydrolysis of the organic material and at least partial conversion of organic N (nitrogen) present in the organic material to inorganic N, and wherein essentially no biogas is produced;

ii) subjecting the organic material fermented in step i) to lime pressure cooking, wherein the lime-pressure cooking results in a further hydrolysis of said organic material, and wherein said lime pressure cooking step results in the conversion of inorganic N to ammonia fluids;

iii) subjecting the organic material fermented in step i) and subjected to lime pressure cooking in step ii) to a strictly anaerobic fermentation resulting in the production of biogas under conditions wherein the pH level of the anaerobic fermenter is kept within a predetermined pH range by contacting or injecting the organic material with fluids comprising CO_2 in amount sufficient to achieve said pH control.

Accordingly, when the first microbial fermentation reaction results in a conversion of organic bound N to inorganic N - an increased amount of biogas can be produced in the second microbial fermentation reaction (i.e. the methanogenesis) - as more inorganic N enters the lime pressure cooking step - where the inorganic N is converted to gaseous ammonia which is stripped. When more nitrogen is stripped during lime pressure cooking, less nitrogen will remain and cause potential problems with ammonia inhibition during the subsequent methanogenesis.

In an exemplary embodiment, provided that the pre-incubation step achieves a convertion of 75 % of the orgN to TAN, then, for a typical poultry litter substrate having 16 g OrgN/kg and 6 g TAN/kg, this would result in increasing the TAN-pool by an additional 75 % of 16 g/kg = 12 g/kg, thereby increasing the removable Nitrogen to 18 g/kg. With Nitrogen stripping of at least 65 % of the TAN-pool during the NiX-treatment, without pre-incubation, 65 % of 6 g TAN/kg = 3.9 g TAN/kg would be removed by stripping, whereas with the pre-incubation step present, the stripped amount increases to 65 % of 18 g TAN/kg = 11.7 g TAN/kg. The overall nitrogen removal per kg of substrate added is therefore in this scenario increased from 3.9 g TAN/kg to 11.7 g TAN/kg. The pre-incubation thus reduces overall the TAN-concentration in the anaerobic digester (biogas fermenter).

The facultative anaerobic bacteria according to the methods of the present invention have a temperature optimum in the range of from approx. 30°C to 37°C. Accordingly, the bacteria can be termed “mesophilic” because of this temperature optimum.

It has also been observed that seeding of the pre-incubation is important for the pre-fermentation which takes place - and approx. 10 to 20 % (w/w) of the contents of a pre-fermentation tank is preferably retained and re-circularized to the next batch pre-fermentation. Accordingly, several interconnected pre-fermenters can be present - so that one can seed approx. 10 to 20 % (w/w) of the contents of one pre-fermenter into a connected pre-fermenter.

Any suitable number, such as fx 2, 3, 4, 5 or 6 interconnected pre-fermentation tanks can be operated as individual, but connected pre-fermentation “batch” fermentations at different stages of the pre-fermentation can be present. Each “batch” pre-fermenter is connected to the lime-pressure cooker and pre-fermented biomass can be diverted from any pre-fermenter to the lime pressure cooker. In this way, one will be able to operate the methods of the present invention as a continuous fermentation process for pre-fermentation and biogas production.

The maximum TS (total dry matter) content of the biomass subjected to pre-fermentation is preferably approx. 30%, such as at the most 25 % (w/w).

The pH optimum for the pre-fermentation is broad and ranges from a pH value of approx. 6.5 to a pH value of approx. 8.5. pH values following a pre-fermentation are preferably in the range of from approx. 6.0 to approx. 7.5.

The duration of the pre-fermentation will depend on the reaction conditions, including temperature, pH, total dry matter content, and the like. It is preferred that the pre-fermentation is at the most approx. 96 hours, such as at the most 72 hours, for example at the most 60 hours, such as at the most 50 hours, for example 40 hours. However, both longer and shorter durations can be employed.

It is possible to obtain, following a pre-fermentation step according to the present invention, a conversion of organic bound N to inorganic N of up to 70 to 80 %. However, depending on the reaction conditions and the content of organic N, lower values can also be obtained – such as for example conversion rates of approx. 35%, approx. 40%, approx. 45%, approx. 50%, approx. 55%, approx. 60%, and approx. 65%. This will also depend on the reaction conditions employed for the pre-fermentation.

Importantly, in one embodiment, at least 80%, such as at least 85%, for example at least 90%, such as at least 95% or more of all nitrogen containing organic acids, such as e.g. uric acid, are converted to ammonia N during a pre-fermentation step operated under the conditions disclosed e.g. herein above.

It is preferred that a minimum of 30%, such as a minimum of 40%, for example a minimum of 50%, such as a minimum of 60% of the organic bound nitrogen originating from protein is converted into inorganic N during a pre-fermentation.

The anaerobic fermentation can in principle be conducted either i) as a pre-incubation step, prior to a nitrogen extraction step, and/or ii) in the form of an anaerobic fermentation and biogas production (methanogenesis, cf. above) conducted following a nitrogen extraction step. This is illustrated in figures 26 to 29. Depending of the retention time of the nitrogen extracted biomass in the buffer tank illustrated in Figures 26 to 29, and the reaction conditions, it is in principle also possible to regard as an anaerobic fermentation the process which takes place while the nitrogen extracted biomass resides in the buffer tank. The aim of the buffer tank of to adjust the

temperature and the pH of the nitrogen extracted (i.e. lime pressure cooked) biomass prior to entry of this biomass into the biogas producing plant.

Acetate and hydrogen produced in the first stages of an anaerobic fermentation can be used directly by methanogens. Other molecules, such as volatile fatty acids (VFAs) with a chain length that is greater than that of acetate, must first be catabolised into compounds that can be directly metabolised by methanogens.

The biological process of acidogenesis is one wherein there is further breakdown of the remaining components by acidogenic (fermentative) bacteria. Here, VFAs are created along with ammonia, carbon dioxide, and hydrogen sulphide, as well as other by-products.

The third stage of an anaerobic fermentation is acetogenesis. Here, simple molecules created through the acidogenesis phase are further digested by acetogens to produce largely acetic acid as well as carbon dioxide and hydrogen.

The final stage of an anaerobic fermentation is that of methanogenesis. Methanogens metabolise intermediate compounds formed during the preceding stages of the anaerobic fermentation, and these compounds are metabolised into methane, carbon dioxide, and water. The afore-mentioned compounds are the major constituents of a biogas.

Methanogenesis is sensitive to both high and low pHs - and methanogenesis generally occurs between pH 6.5 and pH 8.

Remaining, non-digestible organic material that the microbes present in the biogas fermenter cannot metabolise, along with any dead bacterial remains, constitutes what is termed the digestate from the fermentation.

Apart from having a high energy potential, many organic materials also have a high content of nitrogen (N) - in the form of inorganic N (calculated as TAN - total amount of N (NH_3 and NH_4^+)) and organic N e.g. present in proteins, uric acid and other organic sources of N.

When such organic materials are used as substrates for converting organic materials into bio energy in a bio energy plant, in particular biogas in a biogas plant, organic N and / or protein will gradually be converted to ammonia e.g. during an anaerobic fermentation resulting in the production of biogas.

The formation of ammonia in a bio energy plant - especially at high levels - represents a problem as many biogas producing bacteria are sensitive to high levels of ammonia - and high ammonia levels in a biogas fermenter will thus reduce or inhibit the production of methane.

Ultimately, the formation of high levels of ammonia, i.e. above a certain threshold level, cf. below, will kill biogas producing bacteria and inhibit any further biogas formation.

The inhibitory levels of ammonia in a biogas fermenter depend on the conditions used. Under thermophilic fermentation conditions, approx. 3.0 to 4.2 kg ammonia per ton of biomass is considered inhibitory, while under mesophilic fermentation conditions the figure is approx. 5.0 to 7.0 kg ammonia per ton of biomass - depending of the pH value in the digester.

The biogas generating fermentation process can be expected to be completely inhibited at ammonia levels of approx. 7.0 kg to 7.5 kg ammonia per ton of biomass. Accordingly, at this high level of ammonia, fermentation of organic materials by biogas producing bacteria no longer takes place.

It is ammonia (NH_3) which is inhibitory to the biogas production. The equilibrium between ammonia and ammonium (NH_4^+) salts will depend on e.g. pH and temperature. The higher the pH and the higher the temperature, the more the equilibrium is shifted towards the ammonia.

Stripping of ammonia will result in a decreased pH value in the fermenter and it is preferred that the pH value of an anaerobic biogas fermentation shall be below a pH value of approx. 8.5.

The above-cited ammonia inhibition threshold values are generally taken into consideration when operating commercial biogas plants using conventional organic materials as substrates for the biogas producing bacteria. Many such plants are operated according to a two step strategy initially adopting thermophile digestion conditions in a first fermentation step and mesophile digestion conditions in a separate and subsequent, second fermentation step.

The conversion of organic N to ammonia N progresses during an anaerobic fermentation process - i.e. during the process of generating biogas by anaerobic fermentation - and a conversion of as much as approx. 50 % to 70 % of organic N to ammonia N can be expected in accordance with the present invention.

Particular challenges arise when it is desirable to process organic materials having a particularly high N content - as inhibitory levels of ammonia during biogas fermentation can be expected to

occur relatively early on in the fermentation process due to the high levels of organic N and protein in the organic material to be processed.

When employing e.g. poultry manure it is possible to strip up to about 65 % of the ammonia N present in this form of manure. Approximately 30% of all N in poultry manure is in the form of ammonium N. Poultry manure is rich in uric acid and uric acid is not converted - or only converted very inefficiently - to ammonia N during e.g. lime pressure cooking. Hence, there is also a need for devising a strategy for increasing the conversion of organic dry matter, such as uric acid, into ammonium N.

Accordingly, there is a general need for improved methods for organic material processing involving an increased conversion of both total solids (TS) and volatile solids (VS) into ammonium N, as well as securing a biogas production capable of solving the problem of ammonia inhibition of biogas producing bacteria during the production of biogas in a bio energy plant - in particular when more complex types of organic waste products are used – such as e.g. solid manures e.g. from poultry, which have a high organic bound N content.

The present invention thus also provides a technical solution to the problem of how to improve biogas production in a commercial biogas plant. The solution involves novel and inventive methods for reducing organic N contents in an organic biomass material further comprising at least one carbon (C) source during or after the progress of performing an anaerobic fermentation resulting in the production of biogas.

The anaerobic fermentation resulting in the production of biogas may be preceded by one or more initial processing steps aimed at stripping ammonia N from the organic biomass material prior to the biogas production.

One such initial processing step is a pre-incubation step – performed prior to lime pressure cooking – wherein organic N forming part of the biomass to be processed is converted to inorganic N by chemical hydrolysis or by microbial action. The pre-incubation step takes place in a pre-incubation tank, as illustrated e.g. in Figs. 26 to 29.

The pre-incubation step can comprise or be in the form of a facultative, anaerobic fermentation resulting in at least partly converting organic N fractions, including protein N and uric acid N, into an inorganic N fraction which, under suitable conditions, can be stripped as gaseous ammonia by lime pressure cooking, or a processing step functionally equivalent with lime pressure cooking, albeit without subjecting the organic biomass material to a pressure.

Hence, the same effect as can be achieved by lime pressure cooking can also be achieved by simply heating the organic biomass material to a temperature of e.g. above 75°C to 80°C for a longer period of time, but in the absence of applying any pressure above 1 bar.

The pH value of the organic biomass material can be the same irrespective of which alternative one is using. I.e., a pH value of the organic biomass material of preferably above pH = 9 will in principle suffice to provide conditions allowing ammonia to be stripped or extracted.

Accordingly, one initial processing step is that of lime pressure cooking - a step which subjects the optionally pre-incubated organic biomass material, cf. herein above, to an initial hydrolysis under alkaline conditions at an elevated pressure - i.e. more than 1 bar - and at a temperature of more than 100°C. Alternatively, ambient pressure, or a vacuum can also be used for stripping gaseous ammonia from the optionally pre-incubated organic biomass material. It is thus possible to conduct - as an alternative to lime pressure cooking - a step in which the heating of the optionally pre-incubated, organic biomass material takes place at ambient pressure or under vacuum.

Prior to anaerobic digestion and biogas production, yet another pre-treatment step may be used for increasing the conversion of organic bound N in an organic biomass. The lime pressure cooked organic material can be diverted to a buffer tank and the retention time in this buffer tank determines the result of this pre-treatment step. The pH is preferably adjusted to a pH value of less than 8.5, such as less than or about 8.0, for example less than or about 7.5, such as less than or about 7.0, but preferably not less than 6.0. A further conversion and mineralization of organic bound N in an organic biomass can be allowed to occur in the buffer tank and - optionally - ammonia can also be stripped from the buffer tank.

Ammonia N stripped from the organic material e.g. under a lime pressure cooking step - and/or during pre-incubation, and/or during buffer tank treatment - can initially be diverted to a stripper and sanitation tank - or alternatively diverted directly to an absorption column for absorption of the stripped ammonia N. The stripper and sanitation tank will also be connected to an absorption column for absorption of the stripped ammonia N.

According to one presently preferred hypothesis, no organic bound N is converted to ammonia during the lime pressure cooking step. However, organic bound N is converted to ammonia both during the pre-incubation step and during the subsequent anaerobic fermentation resulting in the production of biogas.

Ammonia N stripped from the organic material prior to, during, or after the lime pressure cooking step can be diverted to a stripper and sanitation tank for further incubation under conditions resulting in further conversion of organic N to inorganic N - or, alternatively, diverted directly to an absorption column for absorption of the stripped ammonia N. The absorption column can also be connected to the stripper and sanitation tank so that any ammonia N stripped from the stripper tank can be diverted to the absorption column.

The lime pressure cooked and, at least partly, ammonia N stripped organic material is subsequently diverted to a biogas fermenter and subjected to anaerobic fermentation conditions resulting in the production of biogas. Preferably, the lime pressure cooked organic material is initially diverted to a buffer tank prior to being diverted to the biogas fermenter. Mixing of lime pressure cooked material with further organic materials, for which there is no need for performing a lime pressure cooking step, can take place in a buffer tank prior to diverting the mixture to the biogas fermenter for anaerobic fermentation.

According to this aspect of the present invention, anaerobically fermented organic material is separated into a solid and a liquid fraction. The liquid fraction comprising ammonia N is diverted, or re-cycled, to the lime pressure cooker for stripping of ammonia.

One principle for large scale stripping of ammonia from e.g. a biomass is to increase the pH in combination with aerating and/or heating of the biomass. $Ca(OH)_2$ or CaO, collectively referred to as lime, can be used to increase the pH in a lime pressure cooking step. Lime is used on an industrial scale by for instance the cement industry and is therefore cheap and readily available as a bulk ware. Other bases may also be employed, such as e.g. NaOH or KOH.

When the stripped ammonia is absorbed and an ammonia concentrate is produced, one can divert stripped ammonia to e.g. sulphuric acid present in an absorption column. Sulphuric acid is an industrial bulk ware and it is available in a technical quality appropriate for use in absorption columns stripping ammonia from slurry and other waste waters (e.g. Sacuk et al. 1994).

It is often preferred to strip ammonia by performing a thermal and chemical hydrolysis of a biomass at temperatures of e.g. around or less than 100°C - and at a pressure of about 1 atm. Thermal and chemical hydrolysis of a biomass represents one way to increase the availability of organic material for biogas generation.

Complex carbohydrates, such as e.g. cellulose, hemicelluloses and lignin, are not completely hydrolysed by such treatments. In particular, fibres from straw, maize and other crops are not

made available as suitable substrates for methane formation by such treatments (Bjerre et al 1996; Schmidt and Thomsen 1998; Thomsen and Schmidt 1999; Sirohi and Rai 1998). Accordingly, higher temperatures and a higher pressure will have to be used – and a combination of temperature and pressure will have to be selected depending on the nature of the biomaterial.

In preferred aspects, the present invention concerns methods for performing an anaerobic digestion of a biomass, such as e.g. organic materials comprising one or more of animal manures, energy crops, category 2 waste materials, and similar biomaterials.

Further examples of biomasses capable of being used as an "input biomass" and subsequently processed in accordance with the methods of the present invention are disclosed herein below in more detail. The biomasses can comprise e.g. solid manure waste products from e.g. animal farms, poultry farms, dairies, slaughterhouses, marine fish farms, fish and meat industries as wells as energy crops and or other plants.

The input biomass or feedstock can also comprise liquid manure, dry litter, such as cattle, poultry, offal from cattle, poultry, mink, vegetable oil and glycerin, sludge, whey and the like, corn silage, fish category 2 waste, and industrial waste, including category 3 waste materials.

Biomass can also be any material that comes from plants. Some plants, like sugar cane and sugar beets, store the energy as simple sugars. These are mostly used for food. Other plants store the energy as more complex sugars, called starches. These plants include grains like corn and are also used for food.

Another type of plant matter, called cellulosic biomass, is made up of very complex sugar polymers, and is not generally used as a food source. This type of biomass is under consideration as a feedstock for bioethanol production. Specific feed stocks under consideration include:

1. Agricultural residues (leftover material from crops, such as the stalks, leaves, and husks of corn plants)
2. Forestry wastes (chips and sawdust from lumber mills, dead trees, and tree branches)
3. Municipal solid waste (household garbage and paper products)
4. Food processing and other industrial wastes (black liquor, a paper manufacturing by-product)
5. Energy crops (fast-growing trees and grasses) developed just for this purpose

The main components of these types of biomass are:

- Cellulose is the most common form of carbon in biomass, accounting for 40%-60% by weight of the biomass, depending on the biomass source. It is a complex sugar polymer, or polysaccharide, made from the six-carbon sugar, glucose. Its crystalline structure makes it resistant to hydrolysis, the chemical reaction that releases simple, fermentable sugars from a polysaccharide.

- Hemicellulose is also a major source of carbon in biomass, at levels of between 20% and 40% by weight. It is a complex polysaccharide made from a variety of five- and six-carbon sugars. It is relatively easy to hydrolyze into simple sugars but the sugars are difficult to ferment to ethanol.

- Lignin is a complex polymer, which provides structural integrity in plants. It makes up 10% to 24% by weight of biomass. It remains as residual material after the sugars in the biomass have been converted to ethanol. It contains a lot of energy and can be burned to produce steam and electricity for the biomass-to-ethanol process.

The percentages cited herein below are weight percentages – i.e. (weight / weight), or (mass / mass).

The input biomass has in one aspect of the present invention a carbon content of from 5% to 90% by weight, such as from 5% to 10%, for example from 10% to 15%, such as from 15% to 20%, for example from 20% to 25%, such as from 25% to 30%, for example from 30% to 35%, such as from 35% to 40%, for example from 40% to 45%, such as from 45% to 50%, for example from 50% to 55%, such as from 55% to 60%, for example from 60% to 65%, such as from 65% to 70%, for example from 70% to 75%, such as from 75% to 80%, for example from 80% to 85%, such as from 85% to 90% or any combination of these intervals.

The input biomass has in one aspect of the present invention a protein content of from 5% to 90% by weight, such as from 5% to 10%, for example from 10% to 15%, such as from 15% to 20%, for example from 20% to 25%, such as from 25% to 30%, for example from 30% to 35%, such as from 35% to 40%, for example from 40% to 45%, such as from 45% to 50%, for example from 50% to 55%, such as from 55% to 60%, for example from 60% to 65%, such as from 65% to 70%, for example from 70% to 75%, such as from 75% to 80%, for example from 80% to 85%, such as from 85% to 90% or any combination of these intervals.

The input biomass has in one aspect of the present invention a fat or lipid content of from 5% to 90% by weight, such as from 5% to 10%, for example from 10% to 15%, such as from 15%

to 20%, for example from 20% to 25%, such as from 25% to 30%, for example from 30% to 35%, such as from 35% to 40%, for example from 40% to 45%, such as from 45% to 50%, for example from 50% to 55%, such as from 55% to 60%, for example from 60% to 65%, such as from 65% to 70%, for example from 70% to 75%, such as from 75% to 80%, for example from 80% to 85%, such as from 85% to 90% or any combination of these intervals.

The input biomass has in one aspect of the present invention a nitrogen or ammonia content of from 5% to 90% by weight, such as from 5% to 10%, for example from 10% to 15%, such as from 15% to 20%, for example from 20% to 25%, such as from 25% to 30%, for example from 30% to 35%, such as from 35% to 40%, for example from 40% to 45%, such as from 45% to 50%, for example from 50% to 55%, such as from 55% to 60%, for example from 60% to 65%, such as from 65% to 70%, for example from 70% to 75%, such as from 75% to 80%, for example from 80% to 85%, such as from 85% to 90% or any combination of these intervals.

The input biomass has in one aspect of the present invention a fiber content of from 5% to 90% by weight, such as from 5% to 10%, for example from 10% to 15%, such as from 15% to 20%, for example from 20% to 25%, such as from 25% to 30%, for example from 30% to 35%, such as from 35% to 40%, for example from 40% to 45%, such as from 45% to 50%, for example from 50% to 55%, such as from 55% to 60%, for example from 60% to 65%, such as from 65% to 70%, for example from 70% to 75%, such as from 75% to 80%, for example from 80% to 85%, such as from 85% to 90% or any combination of these intervals.

The input biomass has in one aspect of the present invention a sugar content of from 5% to 90% by weight, such as from 5% to 10%, for example from 10% to 15%, such as from 15% to 20%, for example from 20% to 25%, such as from 25% to 30%, for example from 30% to 35%, such as from 35% to 40%, for example from 40% to 45%, such as from 45% to 50%, for example from 50% to 55%, such as from 55% to 60%, for example from 60% to 65%, such as from 65% to 70%, for example from 70% to 75%, such as from 75% to 80%, for example from 80% to 85%, such as from 85% to 90% or any combination of these intervals.

The input biomass has in one aspect of the present invention a polysaccharide content of from 5% to 90% by weight, such as from 5% to 10%, for example from 10% to 15%, such as from 15% to 20%, for example from 20% to 25%, such as from 25% to 30%, for example from 30% to 35%, such as from 35% to 40%, for example from 40% to 45%, such as from 45% to 50%, for example from 50% to 55%, such as from 55% to 60%, for example from 60% to 65%, such as from 65% to 70%, for example from 70% to 75%, such as from 75% to 80%, for example from 80% to 85%, such as from 85% to 90% or any combination of these intervals.

The input biomass has in one aspect of the present invention a monosaccharide content of from 5% to 90% by weight, such as from 5% to 10%, for example from 10% to 15%, such as from 15% to 20%, for example from 20% to 25%, such as from 25% to 30%, for example from 30% to 35%, such as from 35% to 40%, for example from 40% to 45%, such as from 45% to 50%, for example from 50% to 55%, such as from 55% to 60%, for example from 60% to 65%, such as from 65% to 70%, for example from 70% to 75%, such as from 75% to 80%, for example from 80% to 85%, such as from 85% to 90% or any combination of these intervals.

The input biomass has in one aspect of the present invention a lignin content of from 5% to 90% by weight, such as from 5% to 10%, for example from 10% to 15%, such as from 15% to 20%, for example from 20% to 25%, such as from 25% to 30%, for example from 30% to 35%, such as from 35% to 40%, for example from 40% to 45%, such as from 45% to 50%, for example from 50% to 55%, such as from 55% to 60%, for example from 60% to 65%, such as from 65% to 70%, for example from 70% to 75%, such as from 75% to 80%, for example from 80% to 85%, such as from 85% to 90% or any combination of these intervals.

The input biomass has in one aspect of the present invention a hemicellulose content of from 5% to 90% by weight, such as from 5% to 10%, for example from 10% to 15%, such as from 15% to 20%, for example from 20% to 25%, such as from 25% to 30%, for example from 30% to 35%, such as from 35% to 40%, for example from 40% to 45%, such as from 45% to 50%, for example from 50% to 55%, such as from 55% to 60%, for example from 60% to 65%, such as from 65% to 70%, for example from 70% to 75%, such as from 75% to 80%, for example from 80% to 85%, such as from 85% to 90% or any combination of these intervals.

The input biomass has in one aspect of the present invention a starch content of from 5% to 90% by weight, such as from 5% to 10%, for example from 10% to 15%, such as from 15% to 20%, for example from 20% to 25%, such as from 25% to 30%, for example from 30% to 35%, such as from 35% to 40%, for example from 40% to 45%, such as from 45% to 50%, for example from 50% to 55%, such as from 55% to 60%, for example from 60% to 65%, such as from 65% to 70%, for example from 70% to 75%, such as from 75% to 80%, for example from 80% to 85%, such as from 85% to 90% or any combination of these intervals.

The input biomass has in one aspect of the present invention a sugar polymer content of from 5% to 90% by weight, such as from 5% to 10%, for example from 10% to 15%, such as from 15% to 20%, for example from 20% to 25%, such as from 25% to 30%, for example from 30% to 35%, such as from 35% to 40%, for example from 40% to 45%, such as from 45% to 50%,

for example from 50% to 55%, such as from 55% to 60%, for example from 60% to 65%, such as from 65% to 70%, for example from 70% to 75%, such as from 75% to 80%, for example from 80% to 85%, such as from 85% to 90% or any combination of these intervals.

The input biomass has in one aspect of the present invention a cellulose content of from 5% to 90% by weight, such as from 5% to 10%, for example from 10% to 15%, such as from 15% to 20%, for example from 20% to 25%, such as from 25% to 30%, for example from 30% to 35%, such as from 35% to 40%, for example from 40% to 45%, such as from 45% to 50%, for example from 50% to 55%, such as from 55% to 60%, for example from 60% to 65%, such as from 65% to 70%, for example from 70% to 75%, such as from 75% to 80%, for example from 80% to 85%, such as from 85% to 90% or any combination of these intervals.

The input biomass has in one aspect of the present invention a pH between 0 and 14, such as from 0 to 1, for example from 1 to 2, such as from 2 to 3, for example from 3 to 4, such as from 4 to 5, for example from 5 to 6, such as from 6 to 7, for example from 7 to 8, such as from 8 to 9, for example from 9 to 10, such as from 10 to 11, for example from 11 to 12, such as from 12 to 13, for example from 13 to 14, or any combination of these intervals.

Using virtually any fermentable, organic material as an “input biomass”, including the “input biomasses” cited herein above, the methods of the present invention are capable of producing increased amounts of renewable energy while at the same time refining several nutrients comprised in the digested biomass to fertilizers of commercial quality.

In one embodiment, ammonia stripping results in lowering of the ammonia concentration by more than 10%, such by more than 20%, such as by more than 30%, such as by more than 40%, such as by more than 50%, such as by more than 60%, such as by more than 70%, such as by more than 80%, such as by more than 90%, such as by more than 95% or such as more than 99%.

The level of ammonia and/or nitrogen can be measured before and after the ammonia stripping step and the lowering of the ammonia concentration can be determined.

The ammonia stripping can result in an ammonium concentration of less than 50 g dm^{-3}, such as less than 40 g dm^{-3}, such as less than 30 g dm^{-3}, such as less than 20 g dm^{-3}, such as less than 15 g dm^{-3},such as less than 10 g dm^{-3},such as less than 8 g dm^{-3}, such as less than 6 g dm^{-3}, such as less than 2 g dm^{-3}, such as less than 1 g dm^{-3}, such as less than 0.5 g dm^{-3},or such as less than 0.1 g dm^{-3}.

Anaerobic digestion as used herein shall denote any breakdown of organic matter by bacteria in the absence of oxygen. The terms anaerobic digestion and anaerobic fermentation are used interchangeably herein.

There is provided, in accordance with this aspect of the invention, a method for generating biogas from an anaerobic fermentation of processed organic material comprising solid and liquid parts, said method comprising the steps of

i) subjecting a first organic material comprising one or more organic material sources of organic and inorganic nitrogen to a facultative anaerobic fermentation and converting at least partly said one or more sources of organic nitrogen into inorganic nitrogen,

ii) diverting the anaerobically fermented first organic material comprising one or more sources of nitrogen to a lime pressure cooker;

iii) subjecting said first organic material to a lime pressure cooking step resulting in at least partly hydrolysing said first organic material comprising one or more sources of nitrogen, wherein said lime pressure cooking step results in the formation of ammonia fluids;

iv) diverting said ammonia fluids formed in the lime pressure cooker to an absorption unit;

v) absorbing and condensing ammonia fluids diverted to the absorption unit from the lime pressure cooker;

vi) diverting lime pressure cooked organic material from said lime pressure cooker to a buffer tank and lowering the pH of the organic material in the buffer tank by adding organic and/or inorganic acids to said buffer tank to maintain the pH of the organic material present in the buffer tank preferably within a pH value of from pH = 7.0 to pH = 8.2, or by diverting a CO_2 containing gas, such as biogas, to the organic material of said buffer tank to maintain the pH of the organic material present in the buffer tank within a pH value of from preferably pH = 7.0 to pH = 8.2;

vii) mixing lime pressure cooked organic material with a further organic material in the buffer tank;

viii) diverting the mixed, organic materials to one or more fermenters;

ix) fermenting under anaerobic fermentation conditions said organic materials in said one or more fermenters while maintaining in said one or more fermenters a pH-value of from preferably pH = 7.0 to pH = 8.2 by diverting to the organic material present in each of said one or more fermenters a CO_2 containing gas, such as biogas, in order to maintain the pH of the organic material present in the one or more fermenters within the range of from preferably pH = 7.0 to pH = 8.2, and

x) generating biogas from said anaerobic fermentation of said organic materials.

The above cited method may comprise the further steps of

xi) diverting said organic materials from said one or more fermenters to a separation unit; and

xii) separating organic materials into a solid organic material fraction and a liquid fraction comprising one or more sources of nitrogen.

Additionally, the above cited method may comprise the step of diverting the liquid fraction comprising one or more sources of nitrogen to the lime pressure cooker and/or to the buffer tank. Accordingly, there is also provided the further steps of

xiii) diverting said liquid fraction comprising one or more sources of nitrogen to the lime pressure cooker and/or to the buffer tank, and either

xiv) mixing, in the lime pressure cooker and/ or in the buffer tank, said liquid fraction comprising one or more sources of nitrogen with a first organic material and/or a further organic material comprising one or more sources of nitrogen

wherein, when the mixing takes place in the lime pressure cooker, ammonia originating from said one or more sources of nitrogen present in said liquid

fraction can be stripped from said liquid fraction together with ammonia originating from additional inorganic nitrogen sources present in the first or further organic material, and/or,

wherein, when the mixing takes place in the buffer tank, the pH of the organic material present in the buffer tank will be lowered as a result of the addition of the liquid fraction to the buffer tank.

Accordingly, when the anaerobically fermented and lime pressure cooked organic material is diverted from said lime pressure cooker to a buffer tank, the pH value of the anaerobically fermented and lime pressure cooked organic material can be lowered by preferably contacting the anaerobically fermented and lime pressure cooked organic material with an acid, such as an organic or inorganic acid, or by contacting the pre-incubated and lime pressure cooked organic material with a carbon dioxide (CO_2) containing gas.

The diversion to the buffer tank of CO_2 containing gas, or an organic or inorganic acid, controls the pH of the organic material diverted to the buffer tank. The pH of the organic material diverted to the buffer tank is initially above 8.5 when received from the lime pressure cooker, and it is generally preferred to maintain the pH of the organic material present in the buffer tank within a pH value of from preferably pH = 7.0 to 8.2.

A pH value of from 7.0 to 8.2 is also preferred in the anaerobic digester in which the strictly anaerobic fermentation resulting in the production of biogas is conducted under conditions wherein the pH level is kept within this predetermined pH range.

The one or more sources of nitrogen present in the organic material of the liquid fraction preferably comprise inorganic nitrogen sources, such as ammonium salts.

Solid organic material is preferably diverted to the lime pressure cooker from a reception station suitable for receiving solid organic material, and liquid organic material is preferably diverted to the lime pressure cooker from a reception tank suitable for receiving liquid organic material. Lime is preferably diverted to the lime pressure cooker from a lime storage tank suitable for diverting lime directly to the lime pressure cooker.

Solid and/or liquid organic materials for which there is no need for lime pressure cooking can be diverted directly to the buffer tank and mixed with lime pressure cooked organic material in the buffer tank. It is possible in one embodiment to divert ammonia fluids from the buffer tank

to the absorption unit, prior to diverting said mixed organic materials stripped of ammonia from the buffer tank to one or more fermenters suitable for the production of biogas.

The mixed organic materials are preferably fermented initially in a first fermenter under a first set of fermentation conditions, and subsequently diverted to a second or further fermenter and fermented under a second or further set of fermentation conditions. Preferably, the organic materials are initially fermented under thermophile fermentation conditions and subsequently, in a separate fermentation step, the organic materials are fermented under mesophilic fermentation conditions. The biogas produced by thermophilic and/or mesophilic fermentation is preferably diverted to a gas storage facility operably connected to the one or more fermenters.

Biogas as used herein denotes a renewable, gaseous fuel derived from biological materials that can be used as an energy source instead of fossil fuels, typically to replace conventional natural gas, propane, heating fuel oil, diesel fuel, or gasoline.

Raw biogas is composed of a mixture of combustible gases (principally methane, but also including hydrogen and light hydrocarbons, such as e.g. carbon monoxide, ethane, etc.), and various inert gases and impurities, such as carbon dioxide and hydrogen sulfide. Methane is a combustible gas with the chemical formula CH_4 that can come from fossil or renewable processes.

The present invention can be used for producing increased amounts of biogas from a wide range of organic substrates, including all types of animal manures, energy crops, crops residues and other organic waste materials, including category 2 waste materials.

The present invention is also directed to an optimized waste-to-energy process based on bio-gasification using anaerobic digestion and wet fermentation for increasing the yield of energy obtained e.g. per ton of biomass.

The above-cited method can include a subsequent slurry separation step, i.e. one or more steps resulting in the refinement of selected nutrients, such as phosphor (P) and/or potassium (K) contained in e.g. animal manures. The invention may be applied to separate the main nutrients nitrogen (N) and/or phosphorus (P) from animal manures and refine the nutrients to fertilizer products of commercial quality.

The organic material to be pre-incubated and/or lime pressure cooked can comprise a maximum of 50% solid parts, such as a maximum of 40% solid parts, for example a maximum

of 30% solid parts, such as a maximum of 20% solid parts. The organic material may be in the form of a liquid fraction comprising a maximum of 10% solid parts, or the organic material to be pre-incubated or lime pressure cooked may be mixed with such a liquid fraction.

The lime pressure cooking of the organic material can be performed at a temperature of from more than 100°C to preferably less than 250°C, at a pressure of from preferably 2 to preferably less than 20 bar, and with an addition of lime sufficient to reach a pH value of from about 9 to preferably less than 12, and with an operation time of from at least 10 minutes to preferably less than 60 minutes.

The method may include the step of adding lime (CaO) in an amount of from about 2 to preferably less than 80 g per kg dry matter organic material, such as from about 5 to preferably less than 60 g per kg dry matter.

The methods of the present invention may comprise the step of diverting an organic material to a first fermenter, under a first set of fermentation conditions, and subsequently diverting said fermented, organic material to a second, or further, fermenter, and fermenting said organic material under a second, or further, set of fermentation conditions. The conditions can be thermophile fermentation conditions and/or mesophile fermentation conditions.

The method may include performing the one or more biogas fermentation step(s) at a temperature of from about 15°C to preferably less than about 65°C, such as at a temperature of from about 25°C to preferably less than about 55°C, for example at a temperature of from about 35°C to preferably less than about 45°C.

The fermentation may be allowed to occur over a time of from about 5 days to preferably less than 15 days. The biogas production is achieved by bacterial anaerobic fermentation of the organic material, and the fermentation method may initially performing the biogas production in the first of two plants by anaerobic bacterial fermentation of the organic material, initially by fermentation with thermophilic bacteria in the first plant, followed by diverting the thermophilicly fermented organic material to a second plant, wherein a fermentation with mesophilic bacteria can take place.

Thermophilic reaction conditions include a reaction temperature ranging from 40°C to 75°C, such as a reaction temperature ranging from 55°C to 60°C, whereas a reaction temperature ranging from 20°C to 40°C, such as from 30°C to 35°C is characteristic for a mesophilic fermentation.

In one embodiment, the methods of the present invention results in a biogas production output in Nm^3 per 1,000 tons biomass input of more than 70,000 Nm^3, such as more than 80,000 Nm^3 per 1,000 tons input, for example more than 90,000 Nm^3 per 1,000 tons input, such as more than 100,000 Nm^3 per 1,000 tons input, for example more than 110,000 Nm^3 per 1,000 tons input, such as more than 120,000 Nm^3 per 1,000 tons input, for example more than 130,000 Nm^3 per 1,000 tons input, such as more than 140,000 Nm^3 per 1,000 tons input, for example more than 150,000 Nm^3 per 1,000 tons input, such as more than 160,000 Nm^3 per 1,000 tons input, for example more than 170,000 Nm^3 per 1,000 tons input, such as more than 180,000 Nm^3 per 1,000 tons input, for example more than 190,000 Nm^3 per 1,000 tons input, such as more than 200,000 Nm^3 per 1,000 tons input, for example more than 250,000 Nm3 per 1,000 tons input, such as more than 300,000 Nm^3 per 1,000 tons input, for example more than 400,000 Nm^3 per 1,000 tons input, such as more than 500,000 Nm^3 per 1,000 tons input, for example more than 1,000,000 Nm^3 per 1,000 tons input, such as more than 75,000 Nm^3 per 1,000 tons input, or for example more than 80,000 Nm^3 per 1,000 tons input.

In another embodiment, methods of the present invention results in an electricity output in KWh per 1,000 tons biomass input of more than 200, such as more than 220 KWh per 1,000 tons input, for example more than 240 KWh per 1,000 tons input, such as more than 260 KWh per 1,000 tons input, for example more than 280 KWh per 1,000 tons input, such as more than 300 KWh per 1,000 tons input, for example more than 320 KWh per 1,000 tons input, such as more than 340 KWh per 1,000 tons input, for example more than 360 KWh per 1,000 tons input, such as more than 380 KWh per 1,000 tons input, for example more than 400 KWh per 1,000 tons input, such as more than 450 KWh per 1,000 tons input, for example more than 500 KWh per 1,000 tons input, such as more than 600 KWh per 1,000 tons input, for example more than 700 KWh per 1,000 tons input, such as more than 800 KWh per 1,000 tons input, or for example more than 1,000 KWh per 1,000 tons input.

In a further embodiment, methods of the present invention results in a heat output in MWh per 1,000 tons biomass input of more than 200 MWh per 1,000 tons input, such as more than 220 MWh per 1,000 tons input, for example more than 240 MWh per 1,000 tons input, such as more than 260 MWh per 1,000 tons input, for example more than 280 MWh per 1,000 tons input, such as more than 300 MWh per 1,000 tons input, for example more than 320 MWh per 1,000 tons input, such as more than 340 MWh per 1,000 tons input, for example more than 360 MWh per 1,000 tons input, such as more than 380 MWh per 1,000 tons input, for example more than 400 MWh per 1,000 tons input, such as more than 450 MWh per 1,000 tons input, for example more than 500 MWh per 1,000 tons input, such as more than 600 MWh per 1,000

tons input, for example more than 700 MWh per 1,000 tons input, such as more than 800 MWh per 1,000 tons input, or for example more than 1,000 MWh per 1,000 tons input.

In an even further embodiment, methods of the present invention results in a steam output in MWh per 1,000 tons biomass input of more than 40 MWh per 1,000 tons input, such as more than 50 MWh per 1,000 tons input, for example more than 60 MWh per 1,000 tons input, such as more than 70 MWh per 1,000 tons input, for example more than 80 MWh per 1,000 tons input, such as more than 90 MWh per 1,000 tons input, for example more than 100 MWh per 1,000 tons input, such as more than 105 MWh per 1,000 tons input, for example more than 110 MWh per 1,000 tons input, such as more than 115 MWh per 1,000 tons input, for example more than 120 MWh per 1,000 tons input, such as more than 125 MWh per 1,000 tons input, for example more than 130 MWh per 1,000 tons input, such as more than 150 MWh per 1,000 tons input, for example more than 200 MWh per 1,000 tons input, such as more than 300 MWh per 1,000 tons input, or for example more than 500 MWh per 1,000 tons input.

Bioenergy plant for producing bioenergy - including biogas

In another aspect of the present invention there is provided a plant for generating biogas from an anaerobic fermentation of processed organic material comprising solid and liquid parts, said plant comprising

i) a lime pressure cooker for hydrolysing a first organic material comprising one or more sources of nitrogen;

ii) an absorption unit for absorbing and condensing ammonia fluids diverted to the absorption unit from the lime pressure cooker when said first organic material is subjected to lime pressure cooking;

iii) a buffer tank for mixing lime pressure cooked organic material with a further organic material prior to diverting the mixed, organic materials to one or more fermenters; wherein the buffer tank is optionally fitted with means for diverting a CO_2 containing gas to the organic material present in the buffer tank;

iv) one or more fermenters for anaerobically fermenting said organic materials diverted to said one or more fermenters from said buffer tank, wherein said fermentation results in the generation of biogas, wherein the one or more fermenters for anaerobically fermenting said organic materials diverted to said

one or more fermenters from said buffer tank is/are optionally fitted with means for diverting or re-cycling a CO_2 containing gas to the organic material present in the fermenters in order to maintain a pH value in the fermenters of from preferably pH = 7.0 to pH = 8.2;

v) a separation unit for separating organic materials diverted to the separation unit from said one or more fermenters, wherein said separation of said organic materials results in the generation of a solid organic material fraction and a liquid fraction comprising one or more sources of nitrogen; and

vi) means for diverting said liquid fraction comprising one or more sources of nitrogen to the lime pressure cooker, wherein said liquid fraction is mixed in the lime pressure cooker with first organic material comprising one or more sources of nitrogen.

The lime pressure cooker for hydrolysing said first organic material comprising one or more sources of nitrogen is preferably operably connected to a reception station suitable for receiving solid organic material and/or to a reception tank suitable for receiving liquid organic material. The lime pressure cooker is also in one embodiment operably connected to a lime storage tank suitable for diverting lime directly to the lime pressure cooker.

The absorption unit for absorbing and condensing ammonia fluids diverted to the absorption unit from the lime pressure cooker preferably comprises a steam condenser and a scrubber.

The buffer tank is operably connected to a reception station suitable for receiving solid organic material and/or operably connected to a reception tank suitable for receiving liquid organic material. Also, the buffer tank is in one embodiment further operably connected to the absorption unit for absorbing and condensing ammonia fluids, wherein said connection allows ammonia to be stripped in the buffer tank and diverted to the absorption unit.

The plant according to the second aspect of the invention in one embodiment further comprises a silage tank for storage of energy crops.

The plant comprises one, or more than one, fermenter for anaerobically fermenting said organic materials, wherein said one or more than one fermenter are serially connected so that organic material having been fermented in a first fermenter under a first set of fermentation

conditions can be diverted to a second or further fermenter and fermented under a second or further set of fermentation conditions.

In one embodiment the plant according to the present invention does not contain a stripper and sanitation tank connected to the lime pressure cooker and an absorption unit for absorbing ammonia N. Accordingly, the lime pressure cooker is connected directly to the absorption unit and ammonia formed in the lime pressure cooker during operation thereof under practical circumstances is diverted directly to the absorption unit.

In this embodiment, the lime pressure cooker is also connected to a buffer tank which is connected to the lime pressure cooker and which is not connected to the absorption unit. The buffer tank receives lime pressure cooked biomass from the lime pressure cooker and optionally also biomass which has not been processed in the lime pressure cooker. The buffer tank is connected to one or more biogas reactors. Furthermore, the lime pressure cooker is connected to the one or more biogas reactors and receives from said one or more biogas reactors a liquid fraction comprising ammonia N. The liquid fraction is obtained by removing digestate from liquid biomass removed from the one or more biogas reactors. This can be achieved in a number of ways according to state-of-the-art methods.

The obtained liquid fraction can be diverted or recycled back to the lime pressure cooker where the liquid fraction is stripped for ammonia N without mixing the liquid fraction with a further biomass. Alternatively, the liquid fraction is mixed with a further biomass which enters the lime pressure cooker prior to processing and stripping of ammonia N. Once stripped at least partly for ammonia N, the liquid fraction can be diverted to the buffer tank and mixed with biomass entering this buffer tank directly and without having been subjected to an initial lime pressure cooking step.

In one embodiment, stripping of ammonia N from the liquid fraction takes place only by lime pressure cooking and not by any other means.

It is preferred that the more than one fermenter comprises at least one primary fermenter suitable for thermophilic fermentation and at least one secondary fermenter suitable for mesophilic fermentation. A gas storage facility is operably connected to the one or more than one fermenters.

In one embodiment, the bioenergy plant according to the present invention further comprises biogas fermenters comprising one or more service facilities, or maintenance shafts.

The plant may further comprise a lime pressure cooker for hydrolysing said first organic material comprising one or more sources of nitrogen is operably connected to a reception tank suitable for receiving liquid organic material.

The plant may further comprise a lime pressure cooker for hydrolysing said first organic material comprising one or more sources of nitrogen is operably connected to a lime storage tank suitable for diverting lime directly to the lime pressure cooker.

The plant may further comprise an absorption unit for absorbing and condensing ammonia fluids diverted to the absorption unit from the lime pressure cooker comprises a steam condenser and a scrubber.

The plant may further comprise a pre-incubation tank is operably connected to a reception station suitable for receiving solid organic material, wherein the buffer tank is also operably connected to a reception tank suitable for receiving liquid organic material. The pre-incubation tank can further be operably connected to the absorption unit for absorbing and condensing ammonia fluids, wherein said connection allows ammonia to be stripped in the pre-incubation tank and diverted to the absorption unit.

The plant may further comprise more than one fermenter for anaerobically fermenting said organic materials, wherein said more than one fermenters are serially connected so that organic material having been fermented in a first fermenter under a first set of fermentation conditions can be diverted to a second or further fermenter and fermented under a second or further set of fermentation conditions.

The more than one fermenter preferably comprises at least one primary fermenter suitable for thermophilic fermentation and, serially connected thereto, at least one secondary fermenter suitable for mesophilic fermentation. The plant may further comprise a gas storage facility operably connected to the afore-mentioned, one or more biogas fermenters.

Items of the present invention

Selected items of the present invention are disclosed herein below.

1. A method for generating biogas from an anaerobic fermentation of processed organic material comprising solid and liquid parts, said method comprising the steps of

i) diverting a first organic material comprising one or more sources of nitrogen to a pre-incubation tank and subjecting said organic material to a mineralisation by chemical and/or biological means, wherein the mineralisation results in the conversion of organic N (nitrogen) present in the organic material to inorganic N;

ii) diverting the pre-incubated first organic material to a lime pressure cooker and subjecting said first pre-incubated organic material to lime pressure cooking, wherein said lime pressure cooking step results in the formation of ammonia fluids;

iii) diverting pre-incubated and lime pressure cooked organic material from said lime pressure cooker to a buffer tank and lowering the pH value of the pre-incubated and lime pressure cooked material by contacting the pre-incubated and lime pressure cooked organic material with a carbon dioxide (CO_2) containing gas.

2. The method of item 1 comprising the further step of diverting said ammonia fluids formed in the lime pressure cooker to an absorption unit, and absorbing and condensing the ammonia fluids diverted to the absorption unit from the lime pressure cooker.

3. The method of item 1 comprising the further step of mixing in the buffer tank the lime pressure cooked organic material with a further organic material; wherein the further organic material preferably has not been subjected to lime pressure cooking prior to the mixing with the lime pressure cooked organic material.

4. The method of any of items 1 to 3 comprising the further step of diverting the optionally mixed, organic material(s) from the buffer tank to at least one, anaerobic biogas fermenter suitable for conducting an anaerobic, bacterial digestion of the organic material, wherein the anaerobic, bacterial digestion results in the generation of biogas.

5. The method of item 4 comprising the further step of fermenting, under anaerobic fermentation conditions, the organic material diverted to the anaerobic biogas fermenter, and collecting the biogas resulting from said anaerobic fermentation of said organic material.

6. The method of item 5 comprising the further step of diverting part or all of the organic material from the anaerobic biogas fermenter to a separation unit, and separating the organic material into a solid organic material fraction and a liquid organic material fraction comprising one or more sources of nitrogen selected from organic nitrogen sources and inorganic nitrogen sources.

7. The method of item 6 comprising the further step of diverting said liquid organic material fraction comprising one or more sources of nitrogen to the pre-incubation tank and/or to the lime pressure cooker.

8. The method of item 7 comprising the further step of mixing, in the pre-incubation tank and/or in the lime pressure cooker, said liquid fraction comprising one or more sources of nitrogen with a second organic material comprising one or more sources of nitrogen selected from organic nitrogen sources and inorganic nitrogen sources.

9. The method of item 8 comprising the further step of mineralising the mixture of second organic material and liquid fraction by chemical and/or biological means, when the liquid fraction comprising one or more sources of nitrogen is diverted to the pre-incubation tank and mixed with the second organic material in the pre-incubation tank, wherein the mineralisation results in a conversion of organic N (nitrogen) present in the second organic material to inorganic N.

10. The method of any of items 8 and 9 comprising the further step of stripping ammonia from the mixture of second organic material and liquid fraction, when the liquid fraction comprising one or more sources of nitrogen is diverted to the lime pressure cooker and mixed with the second organic material in the lime pressure cooker.

11. A method for generating biogas from an anaerobic fermentation of processed organic material comprising solid and liquid parts, said method comprising the steps of

    i) diverting a first organic material comprising one or more sources of nitrogen to a pre-incubation tank and subjecting said organic material to a mineralisation by chemical and/or biological means, wherein the mineralisation results in the conversion of organic N (nitrogen) present in the organic material to inorganic N;

ii) diverting the pre-incubated first organic material to a lime pressure cooker and subjecting said first pre-incubated organic material to lime pressure cooking, wherein said lime pressure cooking step results in the formation of ammonia fluids;

iii) diverting pre-incubated and lime pressure cooked organic material from said lime pressure cooker to a buffer tank and lowering the pH value of the pre-incubated and lime pressure cooked material in the buffer tank.

12. The method of item 11 comprising the further step of diverting said ammonia fluids formed in the lime pressure cooker to an absorption unit, and absorbing and condensing the ammonia fluids diverted to the absorption unit from the lime pressure cooker.

13. The method of item 11 comprising the further step of mixing in the buffer tank the lime pressure cooked organic material with a further organic material; wherein the further organic material preferably has not been subjected to lime pressure cooking prior to the mixing with the lime pressure cooked organic material.

14. The method of any of items 11 to 13 comprising the further step of diverting the optionally mixed, organic material(s) from the buffer tank to at least one, anaerobic biogas fermenter suitable for conducting an anaerobic, bacterial digestion of the organic material, wherein the anaerobic, bacterial digestion results in the generation of biogas.

15. The method of item 14 comprising the further step of fermenting, under anaerobic fermentation conditions, the organic material diverted to the anaerobic biogas fermenter, and collecting the biogas resulting from said anaerobic fermentation of said organic material.

16. The method of any of items 1 to 10 and 15 wherein the pH value of the organic material diverted to the anaerobic biogas fermenter is maintained within a predetermined pH-range by contacting the organic material present in the anaerobic biogas fermenter with re-circulated biogas or a biogas diverted to the anaerobic biogas fermenter from an external source, wherein said contacting results in said pH value being maintained within a predetermined pH-range.

17. The method of any of items 14 to 16 comprising the further step of diverting part or all of the organic material from the anaerobic biogas fermenter to a separation unit, and

separating the organic material into a solid organic material fraction and a liquid organic material fraction comprising one or more sources of nitrogen selected from organic nitrogen sources and inorganic nitrogen sources.

18. The method of item 17 comprising the further step of diverting said liquid organic material fraction comprising one or more sources of nitrogen to the pre-incubation tank and/or to the lime pressure cooker.

19. The method of item 18 comprising the further step of mixing, in the pre-incubation tank and/or in the lime pressure cooker, said liquid fraction comprising one or more sources of nitrogen with a second organic material comprising one or more sources of nitrogen selected from organic nitrogen sources and inorganic nitrogen sources.

20. The method of item 19 comprising the further step of mineralising the mixture of second organic material and liquid fraction by chemical and/or biological means, when the liquid fraction comprising one or more sources of nitrogen is diverted to the pre-incubation tank and mixed with the second organic material in the pre-incubation tank, wherein the mineralisation results in a conversion of organic N (nitrogen) present in the second organic material to inorganic N.

21. The method of any of items 19 and 20 comprising the further step of stripping ammonia from the mixture of second organic material and liquid fraction, when the liquid fraction comprising one or more sources of nitrogen is diverted to the lime pressure cooker and mixed with the second organic material in the lime pressure cooker.

22. The method of any of items 6 and 17, wherein at least part of the liquid organic material fraction comprising one or more sources of nitrogen selected from organic nitrogen sources and inorganic nitrogen sources is diverted to the buffer tank, wherein said diversion of said liquid organic material fraction results in lowering the pH-value of the pre-incubated and lime pressure cooked organic material present in the buffer tank.

23. The method of any of items 4 and 14 comprising the further step of diverting to the anaerobic digester a further organic biomass, wherein said further organic biomass is mixed in the anaerobic digester with the optionally mixed, organic material(s) diverted to the anaerobic digester from the buffer tank.

24. The method of item 23, wherein said further organic biomass has not been subjected to lime pressure cooking.

25. The method of any of items 1 to 24, wherein the pH value of the pre-incubated material subjected to lime pressure cooking in the lime pressure cooker is preferably kept, during the lime pressure cooking treatment, within a pH range of from pH = 7.5 to pH = 12.0; for example from pH = 7.5 to pH = 11.5, such as from pH = 7.5 to pH = 11.0; for example from pH = 7.5 to pH = 10.5; such as from pH = 7.5 to pH = 10.0; for example from pH = 7.5 to pH = 9.5; such as from pH = 7.5 to pH = 9.0.

26. The method of any of items 1 to 25, wherein the pH value of the pre-incubated material subjected to lime pressure cooking in the lime pressure cooker decreases over time due to the stripping of ammonia, wherein the initial pH value of the pre-incubated material to which lime has been added, prior to the pressure cooking, is preferably within a range of from more than pH = 9,5 to preferably less than pH = 12.0; such as from more than pH = 10.0 to preferably less than pH = 12, and wherein the terminal pH value of the pre-incubated and lime-pressure cooked material is preferably at the most pH = 9.5.

27. The method of any of items 1 to 26, wherein the pH value of the pre-incubated and lime pressure cooked organic material in the buffer tank, when having been subjected to fluids comprising CO_2 in amounts sufficient to reduced the pH value of the organic material, is preferably kept within a pH range of from pH = 7.0 to pH = 9.5; for example from pH = 7.0 to pH = 9.2, such as from pH = 7.0 to pH = 9.0; for example from pH = 7.0 to pH = 8.7; such as from pH = 7.0 to pH = 8.5; for example from pH = 7.0 to pH = 8.2; such as from pH = 7.0 to pH = 8.0; for example a pH of around 7.5.

28. The method of any of items 26 and 27, wherein the pH value of the pre-incubated and lime pressure cooked organic material present in the buffer tank, after said organic material having been contacted with fluids comprising CO_2 in amounts sufficient to reduced the pH value of the organic material, is at least about 1 pH value lower than the pH value of the pre-incubated and lime pressure cooked organic material present in the lime pressure cooker.

29. The method of item 28, wherein the lowering of the pH value of the lime pressure cooked organic material present in the buffer tank is obtained i) by contacting said organic material in the buffer tank with a CO_2 containing gas, such as biogas, and/or ii) by contacting said organic material in the buffer tank with an acid selected from an organic acid and an

inorganic acid, and/or iii) by diverting, following anaerobic digestion and separation of the fermented, organic material, the separated liquid organic material fraction comprising one or more sources of nitrogen selected from organic nitrogen sources and inorganic nitrogen sources to the buffer tank

30. The method of any of items 1 to 29, wherein the pH value in the anaerobic digester of the pre-incubated and lime pressure cooked organic material subjected to anaerobic digestion is preferably kept within a pH range of from pH = 7.0 to pH = 8.5; for example from pH = 7.0 to pH = 8.2, such as from pH = 7.0 to pH = 8.0; for example from pH = 7.0 to pH = 7.8; such as from pH = 7.0 to pH = 7.5; such as from pH = 7.2 to pH = 8.5; for example from pH = 7.2 to pH = 8.2, such as from pH = 7.2 to pH = 8.0; for example from pH = 7.2 to pH = 7.8; such as from pH = 7.2 to pH = 7.5, for example from pH = 7.4 to pH = 8.5; for example from pH = 7.4 to pH = 8.2, such as from pH = 7.4 to pH = 8.0; for example from pH = 7.4 to pH = 7.8.

31. The method of any of items 1 to 30, wherein, during the pre-incubation step, if any biogas is produced, the amount of biogas constitutes less than 5 %, such as less than 4 %, for example less than 3 %, such as less than 2 %, for example less than 1 % of the amount of biogas produced during the anaerobic digestion step.

32. The method of any of items 1 to 30, wherein percentage conversion of organic N into inorganic N in the per-incubation tank is around at least 35 % of the total organic fraction, such as at least 40%, preferably at least 45%, at least 50%, more preferably at least 55%, at least 60%, even more preferably at least 65%.

33. The method of any of items 1 to 30, wherein the percentage conversion of organic N into inorgnanic N in the incubation tank is around 70% to 80% of the total organic fraction.

34. The method of any of items 1 to 30, wherein, in the pre-incubation tank, nitrogen containing organic acids, such as uric acid, is converted to inorganic N in an amount of 80% or more of the organic acid fraction, such as 90% or more, preferably 95% or more, even more preferably around 100% or more of the organic acid fraction.

35. The method of any items 1 to 30, wherein, in the pre-incubation tank, organic bound nitrogen originating from protein is converted to inorganic N by a minimum of 30% of the organic bound N originating from protein fraction, such as by a minimum of 40%, by a

minimum of 50%, by a minimum of 60% of the organic bound N originating from protein fraction.

36. The method of any of items 1 to 30, wherein conversion of the organic N to inorganic N in the pre-incubation tank is performed at a temperature in the range of approximately 30°C to 37°C, such as from 33°C to 37°C, for example around 37°C.

37. The method of any of items 1 to 30, wherein, when total solid (TS) content of the organic material in the lime pressure cooker is preferably less than approx. 30%, and preferably less than around 25%, such as less than around 23%, the rate of conversion of organic N to inorganic N in the pre-incubation tank is proportional to the amount of water present in the pre-incubation tank.

38. The method of any of items 1 to 30, wherein, for oxygen levels greater than normal oxygen levels in the pre-incubation tank, the rate of conversion of the organic N into inorganic N in the pre-incubation tank is inversely proportional to oxygen level.

39. The method of any of items 1 to 30, wherein, for oxygen levels equal to or lower than the normal oxygen levels in the pre-incubation tank, the rate of conversion of the organic N to inorganic N in the pre-incubation tank is substantially same.

40. The method of any of items 1 to 30, wherein the rate of conversion of the organic N to inorganic N in the pre-incubation tank is directly proportional to the amount of seeding organic material used, wherein the seeding organic material used involves adding material from an active fraction in an amount of approximately 10% to 30% w/w of the active fraction, preferably 10% to 25% w/w, more preferably around 10% to 20% w/w, wherein the active fraction comprises a separate pre-incubated organic material.

41. The method according to item 40, wherein the seeding is selected from adding material by retaining the amount of the active fraction of a first pre-fermentation for subsequent pre-fermentation in the pre-incubation tank; and/ or adding material by receiving, in a first pre-incubation tank, the amount of the active fraction from a second pre-incubation tank.

42. The method of any of items 1 to 30, wherein the step of pre-incubation performed in the pre-incubation tank comprises an operating pH in the pre-incubation tank in the range of around pH = 6.0 to 8.5, typically around pH = 6.4 to 7.5, more typically around pH = 6.5 to 7.2.

43. The method of any of items 1 to 30, wherein the process in the pre-incubation tank comprises an anaerobic facultative microbial fermentation carried out by microbial organisms present in the organic material and optionally also in the seeding material diverted to the pre-incubation tank.

44. The method of any of items 1 to 30, wherein the mineralisation in the pre-incubation tank is performed for around 96 hours or less, such as 72 hours or less, 60 hours or less, 50 hours or less.

45. The method of any of items 1 to 30, wherein the one or more sources of nitrogen in the liquid fraction is an inorganic nitrogen source, such as ammonium.

46. The method of any of the items 1 to 30, wherein the organic material comprises a maximum of 40% solid parts, such as a maximum of 30% solid parts for example a maximum of 25% solid parts such as a maximum of 20% solid parts.

47. The method of any of the items 1 to 30, wherein the percentage of organic N with respect to the total N in the organic material is more than 30%, such as more than 40% w/w, more than 45%, more than 50%, more than 55%, more than 60%, more than 70%, more than 80%.

48. The method of any of the items 1 to 30, wherein the organic material comprises deep litter or manure from animals selected from cattle, pigs and poultry.

49. The method of any of the items 1 to 30, wherein at least part of the uric acid which is present in the organic material is converted into ammonium during the pre-incubation step, and optionally further converted into ammonia, which is optionally collected.

50. The method of any of the items 1 to 30, wherein lime comprises or essentially consists of CaO or $Ca(OH)_2$.

51. The method of any of the items 1 to 30, wherein the amount of added CaO used for lime pressure cooking is from about 2 to about 80 g per kg dry matter, such as from about 5 to about 60 g per kg dry matter.

52. The method of any of the items 1 to 30, wherein the lime pressure cooking of the organic material is performed at a temperature of from about 100°C to preferably less than 250°C, under a pressure of from 2 to preferably less than 20 bar, with addition of lime sufficient to reach a pH value of from about 9 to preferably less than 12, and with an operation time of from at least one 10 minutes to preferably about less than 60 minutes.

53. The method of any of items 1 to 30, wherein said mixed organic materials are fermented in a first, anaerobic fermenter under a first set of fermentation conditions, and subsequently diverted to a second, or further, anaerobic fermenter and fermented under a second or further set of fermentation conditions.

54. The method of any of the items 1 to 30, wherein said organic materials are fermented under thermophile fermentation conditions and/or mesophile fermentation conditions.

55. The method of any of the items 1 to 30, wherein said organic materials are initially fermented under thermophile fermentation conditions and subsequently under mesophile fermentation conditions.

56. The method of any of the items 54 and 55, wherein biogas produced by thermophile and/or mesophile fermentation conditions is diverted to a gas storage facility operably connected to the one or more fermenters.

57. The method of any of the items 1 to 30, wherein the one or more biogas fermentation step(s) is/are performed at a temperature of from about 20°C to preferably less than about 65°C.

58. The method of any of item 57, wherein the thermophilic reaction conditions include a reaction temperature ranging from 40°C to 65°C.

59. The method of any of item 57, wherein the thermophilic reaction conditions include a reaction temperature ranging from 45°C to 60°C.

60. The method of any of item 57, wherein the mesophilic reaction conditions include a reaction temperature ranging from 20°C to 40°C.

61. The method of any of item 57, wherein the mesophilic reaction conditions include a reaction temperature ranging from 32°C to 38°C.

62. The method of any of the items 58 and 59, wherein the thermophilic reaction is performed for about 5 to15 days, such as for about 7 to 10 days.

63. The method of any of the items 60 and 61, wherein the mesophilic reaction is performed for about 5 to 15 days, such as for about 7 to 10 days.

64. The method of any of the preceding items, wherein the nitrogen removal in form of removed inorganic nitrogen with respect to the total nitrogen of the organic material is at least 65%.

65. The method of any of the preceding items, further comprising optionally feeding the anaerobic biogas fermenter with a third organic material for initiating the fermentation of the organic material diverted to the anaerobic fermenter.

66. A method for generating biogas from an anaerobic fermentation of processed organic material comprising solid and liquid parts, said method comprising the steps of

a pre-incubation step comprising receiving in a pre-incubation tank an organic material having a first part removable inorganic N and organic N, and increasing the amount of the removable inorganic N by converting the organic N into a second part of the removable inorganic N using first fermentation conditions, wherein the pre-incubation step is free or at least substantially free from a generation of biogas;

a nitrogen stripping step comprising stripping the removable inorganic N, comprising the first part and the second part, from the pre-incubated organic material using a lime-pressure cooker for stripping said removable inorganic N; and

a second fermentation step comprising anaerobically fermenting, in an anaerobic biogas fermenter, the pre-incubated organic material that is mixed with a further organic material for the generation of biogas

wherein the organic material is contacted with a CO_2 containing gas either following the lime-pressure cooking step and prior to the anaerobic fermentation step, or during the anaerobic fermentation,

wherein the contacting of the organic material with the CO_2 containing gas results of a lowering of the pH of the organic material.

67. A plant for generating biogas from an anaerobic fermentation of processed organic material comprising solid and liquid parts, said plant comprising

i) a pre-incubation tank for mineralisation of an organic material by chemical or biological means; wherein the pre-incubation tank is operably connected to a lime pressure cooker; wherein the mineralisation results in the conversion of organic N (nitrogen) present in the organic material to inorganic N;

ii) a lime pressure cooker for further mineralisation of a first organic material comprising one or more sources of nitrogen;

iii) an absorption unit for absorbing and condensing ammonia fluids diverted to the absorption unit from the lime pressure cooker when said first organic material is subjected to lime pressure cooking;

iv) a buffer tank for mixing lime pressure cooked organic material with a further organic material prior to diverting the mixed, organic materials to one or more fermenters, said buffer tank being operably connected to a gas storage facility suitable for storage of CO_2 containing gas, such as biogas;

v) a gas storage facility suitable for storage of CO_2 containing gas, such as biogas, wherein the gas storage facility is operably connected to the buffer tank;

vi) one or more fermenters for anaerobically fermenting said organic materials diverted to said one or more fermenters from said buffer tank, wherein said fermentation results in the generation of biogas.

68. The plant according to item 67, wherein the plant further comprises

vii) a separation unit for separating organic materials diverted to the separation unit from said one or more fermenters, wherein said separation of said organic materials results in the generation of a solid organic material fraction and a liquid fraction comprising one or more sources of nitrogen; and

viii) means for diverting said liquid fraction comprising one or more sources of nitrogen to the lime pressure cooker, wherein said liquid fraction is mixed in the lime pressure cooker with first organic material comprising one or more sources of nitrogen.

69. The plant according to any of items 67 and 68, wherein the absorption unit for absorbing and condensing ammonia fluids diverted to the absorption unit from the lime pressure cooker comprises a steam condenser and a scrubber.

70. The plant according to any of items 67 and 68, wherein the buffer tank is operably connected to a reception tank or a reception station suitable for receiving liquid and solid organic material, respectively.

71. The plant according to any of items 67 and 68, wherein the buffer tank is further operably connected to the absorption unit for absorbing and condensing ammonia fluids, wherein said connection allows ammonia to be stripped in the buffer tank and diverted to the absorption unit.

72. The plant according to any of items 67 and 68 comprising more than one fermenter for anaerobically fermenting said organic materials, wherein said more than one fermenters are serially connected so that organic material having been fermented in a first fermenter under a first set of fermentation conditions can be diverted to a second or further fermenter and fermented under a second or further set of fermentation conditions.

73. The plant according to any of items 67 and 68, wherein said more than one fermenter comprises at least one primary fermenter suitable for thermophilic fermentation and at least one secondary fermenter suitable for mesophilic fermentation.

74. The plant according to any of items 67 and 68 further comprising a biogas storage facility operably connected to the one or more fermenters.

Examples

The following examples are illustrative of some preferred embodiments of the present invention and these examples should not be interpreted so as to limit the scope of the present invention.

The following abbreviations are generally used in the present examples

AD	-	Anaerobically digested
BMP	-	Biomethane potential
CSTR	-	Continuously stirred tank reactor
HRT	-	Hydraulic retention time
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

Methods for determining nitrogen (N) fractions of an organic material

Nitrogen present in an organic material, such as e.g. layer manure, such as chicken litter, can be divided into different fractions which may be determined individually either by analysis or by calculation.

The total amount of nitrogen (TKN) is made up of inorganic nitrogen (TAN) and organic nitrogen (OrgN).

The TAN fraction is made up of ammonia (NH_3) and ammonium (NH_4^+).

Other inorganic nitrogen containing compounds, such as nitrate (NO_3^-), are usually present in such small amounts that they are considered insignificant.

The OrgN fraction is made up of two sub-fractions: Protein bound nitrogen and uric acid bound nitrogen.

Other nitrogen containing organic compounds, such as DNA and RNA, are usually present in such small amounts that they are considered insignificant.

The above-cited fractions can be determined by the below-cited analysis methods:

TKN can be determined by the Kjeldahl procedure (ISO 5663/DIN EN 25 663)

TAN can be determined by titration (ISO 5663/DIN EN 25 663)

Uric acid nitrogen can be determined by HPLC (Pekic et al.; Chromatographia; Vol. 27; No. 9/10; May 1989)

The following fractions can be determined by calculation:

OrgN is determined as OrgN = TKN – TAN

Protein bound nitrogen is determined as protein bound nitrogen = OrgN – Uric acid nitrogen

Example 1

The following example briefly summarizes pH neutralisation experiments performed on lime pressure cooked biomaterials.

Selected abbreviations used in Example 1

AD	-	Anaerobic digestion
OrgN	-	Organic Nitrogen
RSD	-	Relative standard deviation
SD	-	Standard deviation
TAN	-	Total Ammonium Nitrogen
w/w%	-	Weight percentage
NiX	-	Nitrogen extraction by lime pressure cooking

Summary

As part of a processing step in which a biomaterial is subjected to lime pressure cooking, the pH of the treated material is raised using burnt lime (CaO). This facilitates a reduction in the TAN pool by removal of ammonia and this is a necessary step in order to obtain high removal efficiencies (see Equation 3 and Equation 4).

$$CaO_{(s)} + H_2O_{(l)} \leftrightarrow Ca^{2+}_{(aq)} + 2OH^-_{(aq)}$$

Equation 3

$$2NH^+_{4\,(aq)} + 2OH^-_{(aq)} \leftrightarrow 2NH_{3(g)} + 2H_2O_{(l)}$$

Equation 4

A disadvantage to this method is the low solubility of $CaO_{(s)}$, which results in substantial amounts of residual $CaO_{(s)}$ after the NiX treatment. The surplus base will subsequently be dissolved in the anaerobic digester leading to undesirably high pH levels.

The equilibrium between ammonia ($NH_{3(g)}$) and ammonium (NH_4^+) is pH dependent, and more ammonia will be present with higher pH levels. Since ammonia is the toxic part of the TAN pool, the residual base in the digester counteracts the benefits of the TAN removal obtained during NiX treatment.

Another disadvantage is the potential build-up of calcium ions (Ca^{2+}) which may be detrimental to the stability of the anaerobic digester. It is well known that anaerobic digestion is sensitive to the osmotic potential. However, very little has been published in the litterature with respect to the effect of the concentration of Ca^{2+} on the stability of an anaerobic digester.

The purpose of the investigations reported here was to determine if the pH of NiX treated materials could be lowered by bubbling carbondioxide ($CO_{2(g)}$) through the NiX treated material. It is well known that $CO_{2(g)}$ can be converted to carbonic acid ($H_2CO_{3(aq)}$) if it reacts with water, which in turn could lead to neutralisation of the exogenous base.

The investigations showed that it is possible to lower the pH using $CO_{2(g)}$, and that calcium is precipitated as calcium carbonate ($CaCO_{3(s)}$) during the neutralisation reaction. As detailed in

the Discussion section, the driving force for the reaction is likely the precipitation of $CaCO_{3(s)}$, which continuously removes the produced carbonate.

This mechanism also explains why the pH can be lowered to a more acidic level than in the original substrate. Since a large part of the hydroxyl ions (OH^-) have already been neutralised during TAN removal, the residual Ca^{2+} has the potential to keep driving the production of $H_2CO_{3(aq)}$ past the original pH level. Since the ammonia fraction of the TAN pool is pH dependent, a major potential advantage of acidifying the NiX treated material is that it creates the possibility of reducing the amount of material that needs to be NiX treated.

Results

The experimental setup consisted of

1. A pressurised gas cylinder with a pressure reduction valve
2. A flow meter for measuring and controlling gas flow.
3. Connective tubing
4. A container with NiX treated material

Gas from the pressurised cylinder was bubbled through the NiX treated material via the connective tubing. The flow was controlled and monitored using the reduction valve and the flow meter. The NiX treated material was continously mixed and the pH measured at regular intervals.

Table 1 shows the details of the netralisation set-up parameters.

	NiX treated material	TS	pH (start)	pH (end)	CO_2 (%)	Gas flow	Duration
Neutralisation setup	27 kg	14.6% (± 1.3)	8.5	7.75	100%	1 ml/min	60 min

Table 1 – Neutralisation set-up.

Fig. 25 illustrates the pH development during the experiment referred to in Table 1 above.

In addition to creating a pH drop, there is a significant production of a grainy material during pH neutralisation (see Fig. 64) which was analysed to consist of 99% limestone ($CaCO_{3(s)}$).

Discussion

The pH of NiX treated material was succesfully lowered from 8.5 to 7.75 using $CO_{2(g)}$. This value was chosen to ensure a suitably low pH value in the biogas reactor while disturbing the buffer systems of the NiX material as little as possible. However, other experiments have shown that it is possible to lower the pH to at least 7.2, which is significantly lower than the pH of the original material prior to base addition and NiX treatment (pH 7.7). It has also been shown that pure $CO_{2(g)}$ may be substituted with biogas (~ 40% $CO_{2(g)}$), although with longer neutralisation times.

The precipitated limestone during neutralisation provides an explanation to the mechanism behind the pH drop. It is well established that $CO_{2(g)}$ is in equilibrium with carbonic acid ($H_2CO_{3(aq)}$). The reaction mechanism can be seen in Equation 5

$$CO_{2(g)} + H_2O_{(l)} \leftrightarrow H_2CO_{3(aq)} \leftrightarrow 2H^+ + CO_3^{2-}$$

Equation 5

The production of H^+ (protons) will cause a drop in pH. This reaction will continue as long as either of the reaction products is removed from the reaction.

In the case of the pH neutralisation experiment, the solution contains large amounts hydroxyl ions and calcium ions, due to the addition of burnt lime during the NiX treatment.

When the reaction products of Equation 5 come into contact with the residual components from the NiX treatment the following reaction will take place:

$$2H^+ + CO_3^{2-} + Ca^{2+} + OH^- \leftrightarrow 2H_2O + CaCO_{3(s)}$$

Equation 6

When the newly formed protons from carbonic acid react with hydroxyl ions the result is a lowering of the pH. When calcium ions react with the carbonate ion, the result is limestone which precipitates out of solution due to the low solubility of this product. The continuous

removal of carbonate will allow for more carbonic acid to be formed, and hence the production of more protons.

It seems plausible that the driving force for the reaction is the presence of calcium ions in solution, which will facilitate the production of carbonic acid and hence neutralisation of the pH.

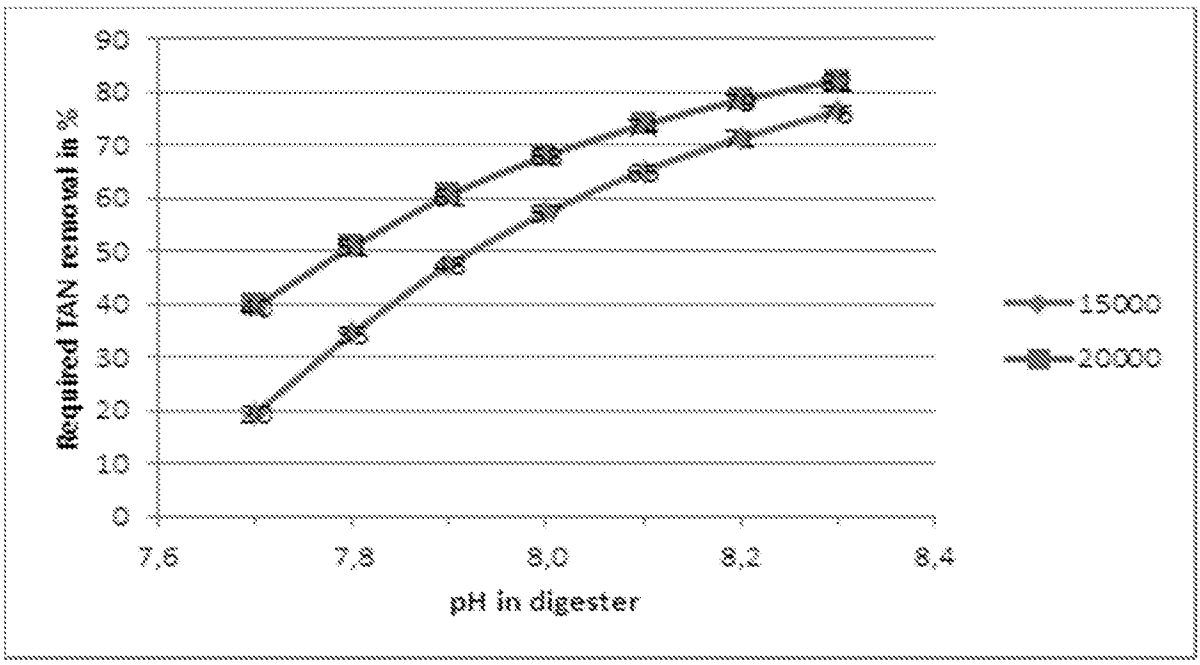
Example 2

The below graph illustrates the requirement for TAN removal (%) - in order to maintain the level of gaseous ammonia in the anaerobic digester below 700 mg per litre - as a function of the pH value of the fermented biomaterial. The value of 700 mg NH_3 per litre is defining a threshold value for NH_3 inhibition and it is thus desirable not to exceed this value

Two different scenarios are represented in the below illustration. In Scenario 1, the contents of total ammonia N (TAN) in the fermenter is 15,000 mg per litre, whereas in Scenario 2, an amount of total ammonia N (TAN) of 20,000 mg per litre is present in the fermenter.

The temperature is kept at 37°C and the pH interval of from pH = 7.7 to pH = 8.3 is illustrated.

By decreasing the pH value as indicated herein below it is possible to reduce the amount of TAN it is necessary to remove so that the threshold value of 700 mg NH_3 per litre is not exceeded.



Example 3

This example summarizes experiments performed on hen litter from caged egg layers. The purpose of the investigations was to

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

Hen litter was sampled on-site and received in 10x100L containers in May 29th 2012 and was set up for BMP analysis on the 11th of June 2012. At the same time the hen litter was analyzed for TS/VS and nitrogen content. BMP assays were run thermophilically for 94 days. The conclusions from the analysis may be seen from Table 2.

Parameter	Unit	Hen Litter
BMP	Nml CH_4/g VS	293 (± 9)
(Untreated)	*Nml CH_4/g wet weight*	*137*(± 5)
BMP	Nml CH_4/g VS	292 (± 6)
(with NiX treatment)	*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 2 – 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 methane yield 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 BMP

The expected methane yield, when digesting the treated and untreated hen litter in a two-stage CSTR setup, may be seen in Figure 26. The calculation combines the digestion speeds and final methane yields obtained in batch to predict the methane production in a two-stage thermophilic/mesophilic system with a 15 days retention time in each reactor. In this setup approx. 91% of the batch BMP value may be realised. Also, there is no effect of NiX treatment on methane yield in CSTR.

1 Materials and Methods

1.1.1 Dry Matter

Total solids (TS) and volatile solids (VS) contents of the hen 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.

1.1.2 Substrate information

Substrate name	**Hen Litter**
Substrate description	Litter from egg layers
Production type	Cages
Sample date	May 2012
Substrate age (at time of sampling)	< 1 day
Sampled by	APJE
Supplier information	Stud Farm, Rufford, Newark-on-Trent, Nottinghamshire, NG22 9HB

Table 3 – Substrate, sampling and supplier information

1.1.3 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 destillations were performed on a Büchi K355 distillation unit

1.2 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 °C. After treatment pressure was released

over a period of 20-30 minutes. Samples were collected and processed according to the flowchart below.

Pressure	Temperature	Pressure hold time	Pressure release time	Sample TS	Sample VS
4 barg	146 °C	20 min	20-30 min.	30%	23%

Table 4 - Pressure cooking parameters

The following process illustrates an overview of the treatment

Hen litter + CaO + H_2O

↓ Pressure cooking

Sampling

↓ Homogenisation

BMP analysis

Two independent Nix treatments were performed on the hen litter.

1.3 Biological Methane Potential (BMP) Test

1.3.1 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 glass bottles. The inoculum was taken from the thermophilic main digester of Foulum biogas plant and incubated at 52 ± 1 °C 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 was used per bottle.

To avoid inhibition due to organic overloading, the BMP assay was carried out at two different concentrations of substrate. Each batch bottle was prepared by addition of either 0.9 or 1.7 g VS followed by addition of 200 ml inoculum. Resulting substrate VS concentration in each of the substrate batch bottles was 4.5 and 8.5 g substrate VS/L inoculum respectively. For each of the two different substrate concentrations 3 replicates were incubated.

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 °C in a heat cabinet for the duration of the batch test.

1.3.2 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 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.

2 Results

2.1 Dry matter analysis

The results of the TS and VS analysis are shown in table 5, below.

Parameter	Unit	Hen Litter
Total Solids	w/w%	46.6 (± 0.9)
Volatile Solids	w/w%	33.9 (± 0.5)

Table 5 – TS and VS levels of Retford Hen Litter.

2.2 Nitrogen analysis

The hen litter was analysed for total ammonium nitrogen (TAN) and total nitrogen (TKN). Table 6 below, shows the content of TAN and TKN in the substrate. Organic nitrogen (OrgN) is calculated by subtraction of TAN from TKN.

Parameter	Unit	Hen Litter
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.

2.3 BMP analysis

The methane production curves from the two different substrate concentrations showed no significant difference, meaning that there is no observable inhibition at the concentrations used in this assay. The data points from the two concentrations are averaged and used as one.

Specific methane yield B(t), is shown in Figure 28 for both untreated hen litter and NiX treated hen litter. Empirical data are shown as points and the best-fit curve from non-linear analysis of the empirical data are shown as lines. For details of the best-fit curve with calculated kinetic constants, please refer to Table 7, below.

The curve for the untreated hen litter shows a steady increase in methane production until a maximum yield of 293 (± 9) Nml CH_4/g VS is obtained after about 30-40 days. The curve for the NiX treated hen litter is almost identical to the untreated, and shows a steady increase in methane production until a maximum yield of 292 (± 6) Nml CH_4/g VS is obtained.

	Retford Hen Litter - Untreated	Retford Hen Litter - NiX
BMP - First order equation		
Best-fit values		
B	293 Nml CH_4/g VS	292 Nml CH_4/g VS
K	0.21	0.22
Std, Error		
B	4.1	2.9
K	0.014	0.01
95% Confidence Intervals		
B	285 to 302	287 to 298
K	0.19 to 0.24	0.20 to 0.24
Goodness of Fit		
Degrees of Freedom	52	106
R^2	0.96	0.96

Table 7 – Details of best-fit analysis of untreated and NiX treated hen litter

Example 4

1. Introduction

In this example, 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 Nm^3 CH_4 per ton VS. Realistic yields per ton of wet weight is therefore at the same level or higher for chicken litter (100-150 Nm^3 CH_4) than for most energy crops (often less than 100 Nm^3 CH_4).

However, chicken litter also has a high nitrogen (N) content, which may 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.

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.

2. Procedure

The investigation was carried out in a pilot scale plant during the period from November 2011 to July 2013. The test was based on a single step digestion at 37°C with hydraulic retention times as specified below for each of the phases.

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 (NH_3) 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) NH_3 concentration in digester (TAN/pH)
3) TS in digester and recycled liquid / water balance
4) Concentration of other substances
5) Energy balance

KPI 1) and 2) were primary as the NH_3 concentration is expected to be the main potential inhibitor of the methane yield. When stable NH_3 concentration and a stable and high methane yield is obtained the focus changes to also include the remaining KPIs.

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

3 Results

3.1 Phase 1. November 2011 to June 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 to initially 34 days and later 29 days. This 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.

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

Methane yield (Figure 29) was stable around 300 L CH_4/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 CH_4/kg VS was reached. This was followed by unstable peaks of higher yields leading to a sudden drop to less than 200 L CH_4/kg VS at the end of the period.

The concentration of TAN (Figure 30) 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.

NH_3 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 31). NH_3 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 NH_3 to return to the previous peak value at nearly 1,100 mg/L.

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 32 and Figure 33). 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 hundred mg/L.

The TS content in the digester (Figure 34) 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 %.

3.1.2 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 35) immediately started to go up from less than 200 L CH_4/kg VS and peaked within a few days at +260 L CH_4/kg VS followed by a very rapid drop during a week to less than 100 L CH_4/kg VS. This level continued for three weeks after which the gas yield increased dramatically to +560 L CH_4/kg VS during the last 3 weeks of the period.

The concentration of TAN (Figure 36) 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.

NH_3 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 37). Water dilution reduced NH_3 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.

The VFA levels (Figure 38) 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.

The longer-chained VFA's showed different trends during the period (Figure 39).

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.

The TS content in the digester (Figure 40) 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 started decreasing and reaching 6.9 % before the end of the period.

3.1.3 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 41) immediately began to drop from the peak level of more than 560 L CH_4/kg VS at the end of Period 1B to around 300 L CH_4/kg VS three weeks later and finally stabilizing at 250 L CH_4/kg VS after 6 weeks.

The concentration of TAN (Figure 42) 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. NH_3 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 43). Consecutively, NH_3 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.

The composition of the VFA pool (Figure 44) 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.

The longer-chained VFA's showed different developments during the period (Figure 45).

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.

The TS content in the digester (Figure 46) 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.

3.1.4 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 breakdown.

Methane yield (Figure 47) was quite constant during the entire period around a mean of 270 L CH_4/kg VS but with drops to 230-240 L CH_4/kg VS several times. However, at the end of the period the yield showed an increasing tendency towards a yield level around 300 L CH_4/kg VS.

The initial concentration of TAN (Figure 48) at 5,200 mg/L increased during the first 2 weeks to a final level of 5,600 mg/L.

NH_3 increased from 700 to 1,200 mg/L during the first week due to increasing pH from 8.1 to 8.3 (Figure 49). A week later a stable NH_3-level at 1,100 mg/L had been reached while the pH had stabilized at around 8.2.

The VFA pool (Figure 50) 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.

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 1,300-1,8000 mg/L, respectively (Figure 51).

The TS content in the digester and the recycled liquid (Figure 52) 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.

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

At the end of Phase 1, it was clear that with TAN levels around 6-7000 ppm 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.

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, but the majority of this is mineralised during digestion and released as TAN in the reactor. To lower the amount of TAN in the reactor, a novel concept was thus developed to shift the nitrogen pool from organically bound to inorganic 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. In summary, nitrogen mineralisation comprises incubation of the chicken litter with an appropriate mixture of liquid from separated digestate with a microbially active culture at 36°C. The active culture 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 to 8.2.

The development of TAN, NH_3 and pH during Phase 2 may be seen in Figures 53 and 54. As may be seen from the figures, pH and ammonia levels were steadily increasing and reached inhibitory levels after 1½ months with no signs of stabilising. The trial was therefore aborted before it was possible to determine where TAN levels would settle.

3.3 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. Although mineralisation allowed stripping of significantly higher amounts of nitrogen, TAN levels in the reactor had not reached equilibrium when Phase 2 was terminated. Moreover, pH was higher than 8.2, meaning that the buffer capacity of the digester was not strong enough to maintain pH at the desired level.

Two additional measures were thus undertaken to reduce the TAN content and the pH level in the digester.

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

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 pH<7 (compared to pH 8.5 to 9.0 without pH neutralisation). 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 55 to Figure 58.

As can be seen from Figure 55, TAN levels increase at a slower rate than in Phase 2 (see Figure 53). 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 are close to reaching equilibrium, the major contributor to the ammonia level is pH.

As can be seen from Figure 56, 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.

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 condition – standard temperature and pressure (273 K and 1 bar). Expected methane production is calculated from a batch BMP test taking into account the retention time in the digester.

As seen from Figure 57 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. This 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 buildup 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.

Although the VFA development has not stabilised yet (Figure 58), 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. During the last two weeks there has been a decrease in acetate and an increase in propionate. As it is common for propionate to tail acetate development the next few weeks will show whether the VFA's are reaching an equilibrium state.

The amount of total solids (Figure 59) 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 methane percentage (Figure 60) in the reactor seems to have stabilised around 60-65%.

4 Discussion

4.1 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 CH_4/kg VS coincides with [NH_3] increasing to more than 1,000 mg/L. This could be seen as a confirmation of the expectation that a threshold value for NH_3 inhibition exists around this level. 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 CH_4/kg VS. Again the drop in methane yield occurred after a peak in [NH_3] which went above 1,000 mg/L just before the start of the period. The gas yield

stayed below 100 L CH_4/kg VS for more than three weeks while the VFA's skyrocketed to more than 35,000 ppm with unusually high contributions from the longer-chained VFA's. With [NH_3] 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 CH_4/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 NH_3 concentrations ranging from just below 1,000 to more than 2,000 mg/L. The high NH_3 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 NH_3 concentrations could be an explanation for the observed stable methane yield.

During period 1D a medium-level OLR was maintained and methane yield were quite stable during the entire period at a level around 270 L CH_4/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 [NH_3] 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 NH_3-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.

4.2 Phase 2

Prior to the initiation of Phase 2 effort was put into developing a method for ensuring an acceptable NH_3 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 ammonia concentrations 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 NH_3, 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.

4.3 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 included in patent application no. PA 2013 00260, which is now incorporated herein by reference.

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 to 9.0.

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.

EXAMPLE 5

The example summarizes experiments 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 for production of biogas. 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 which has been subjected to nitrogen mineralisation.
- Broiler litter which has been subjected to nitrogen mineralisation and NiX treatment.
- Broiler litter which has been subjected to 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 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 center, where it was homogenised and stored. After each treatment samples were collected and analysed.

Key results are presented below in Table 8.

	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 8 - Overview of key results from BMP and TS/VS analysis of broiler litter.

- Untreated litter from broiler chickens reached a final BMP yield of 300 NmL CH_4/g VS, and is comparable to previous analysis of hen litter (293 NmL CH_4/g VS) in Example 3.

- 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 (see Table 15). However, in the second BMP setup there is no significant change in neither final yield nor digestion speed.

- 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.

- pH adjusted material shows an increase in both digestion speed and ultimate yield.

The expected methane yield, when digesting the untreated and treated samples in a two-stage CSTR setup, may be seen in Figure 61. 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.

1. Materials and Methods

1.1.1 Dry Matter

Total solids (TS) and volatile solids (VS = organic dry matter) contents of the broiler 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.

1.1.2 Substrate information – deep litter from suppliers of bull calves

Substrate name	**Chicken litter**
Substrate description	Litter from broilers. 0.8 tons of straw/1.7 tons of wood chips/27.5 tons of chicken faeces
Production type	Stables (25400 birds)
Sample date	November 2012
Substrate age (at time of sampling)	42 days
Sampled by	SBG
Supplier information	Mr Peter McWilliams 56 Tullyaran Road Donaghmore Dungannon Co. Tyrone BT70 3HL Northern Ireland

Table 9 – Substrate, sampling and supplier information

1.2 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 temperature and/or pressure to 4 barg (corresponding to 146°C) or 0 barg (corresponding

to 100 °C). After treatment pressure was released over a period of 20-30 minutes. Samples were collected and processed according to the flowchart below.

Pressure	Temperature	Pressure hold time	Pressure release time	Sample TS	Sample VS	pH after treatment
4 barg	146 °C	20 min	20-30 min.	25%	7.3%	~9.2
0 barg	100 °C	15 min	15-20	25%	7.3%	~9.2

Table 10 – Pressure cooking parameters.

The process giving an overview of the treatment is shown in Example 3, where (chicken litter + CaO + H2O) is pressure cooked for sampling and then homogenized for BMP analysis.

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

1.3 Biological Methane Potential (BMP) Test

1.3.1. 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°C 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.

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°C in a heat cabinet for the duration of the batch test.

1.3.2 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.

2. Results

2.1 TS and VS analysis

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

	Units	Untreated broiler litter
TS	w/w %	64% (±<0.1)
VS	w/w %	53% (±<0.5)

Table 11 – TS and VS levels of broiler litter

2.2 Nitrogen analysis

The broiler litter was analysed for total ammonium nitrogen (TAN) and total nitrogen (TKN) in the untreated and treated samples. Table 12 below, shows the content of TAN and TKN in the substrate. Organic nitrogen (OrgN) is calculated by subtraction of TAN from TKN.

	Units	Broiler litter (untreated)
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 12 – Overview of key results associated with the analysis of broiler litter. Organic nitrogen is calculated by subtraction of total ammonium nitrogen from total nitrogen.

2.3 BMP analysis

Specific methane yields obtained during the BMP analysis of untreated and treated broiler litter are shown in Figure 62 and Figure 63.

The specific methane yield (mL methane per gram substrate VS added) is calculated by subtraction of background and normalizing according to VS concentration.

The broiler litter was analysed for biomethane potential and compared to litter subjected to a combination of 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 mineralisation, NiX treatment at 0 barg and pH adjustment.

	Units	Untreated	Mineralised	Mineralised, NiX treated (4 barg)
BMP	NmL CH_4/g VS *NmL CH_4/g wet weight*	303 (±6) *194 (±4)*	288 (±6)	279 (±4)

Table 13 – Summary of BMP values obtained in the first BMP setup.

	Units	Untreated	Mineralised	Mineralised, NiX treated (0 barg)	Mineralised, NiX treated (0 barg, pH adjusted)
BMP	NmL CH_4/g VS *NmL CH_4/g wet weight*	296 (±9) *189 (±5)*	311 (±9)	297 (±6)	307 (±7)

Table 14 – Summary of BMP values obtained in the second BMP setup.

The digestion profiles for the two BMP setups may be seen in Figure 62 and Figure 63. In both graphs, data points represent empirical data, and solid lines represent the best-fit curve.

Although the absolute methane potential is increased, even if slightly, the digestion speeds vary (compare k-values in Table 15 and Table 16 below).

Best-fit values	Moy Park - Untreated	Moy Park - Mineralised	Moy Park - NiX 4 barg
B	303	288	279
K	0.129	0.0968	0.158
Std. Error			
B	6,05	6,04	3.76
K	0.00943	0.00693	0.00824
Lower 95% conf. limit			
B	291	276	271
K	0.110	0.0829	0.142
Upper 95% conf. limit			
B	315	300	286
K	0.148	0.111	0.175
Goodness of Fit			
Degrees of Freedom	58	58	118
R^2	0.935	0.942	0.933
Absolute Sum of Squares	50667	43273	86847
Sy.x	29.6	27.3	27.1
Constraints			
B	B > 0.0	B > 0.0	B > 0.0
K	K > 0.0	K > 0.0	K > 0.0
Number of points Analyzed	60	60	120
Outliers (excluded, Q=1.0%)	0	0	0

Table 15 – Non-linear best-fit analysis from BMP setup 1 with untreated, mineralised and NiX treated chicken litter.

Best-fit values	Broiler litter - Untreated	Broiler litter - Mineralised	Broiler litter - NiX (0 barg)	Broiler Litter - pH adjusted	Cellulose
B	296	311	297	307	398
K	0.125	0.107	0.129	0.16	0.142
Std. Error					
B	9,42	8,49	6,07	6,67	11,7
K	0.0121	0.00839	0.00812	0.0116	0.0133
Lower 95% conf. Limit					
B	277	293	285	294	375
K	0.101	0.0899	0.113	0.136	0.116
Upper 95% conf. Limit					
B	315	328	309	321	422
K	0.149	0.124	0.146	0.183	0.169
Goodness of Fit					
Degrees of Freedom	40	40	82	40	40
R^2	0.931	0.955	0.941	0.961	0.939
Absolute Sum of Squares	38045	27076	66520	22896	64417
Sy.x	30.8	26	28,5	23.9	40.1
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	42	42	84	42	42
Outliers (excluded, Q=1.0%)	0	0	0	0	0

Table 16 Non-linear best-fit analysis from BMP setup 2 with untreated, mineralised, NiX treated and pH adjusted chicken litter

Example 6

This example illustrates exemplary distributions of various nitrogen (N) containing fractions of a layer manure biomass material which are present at various processing steps when the methods of the present invention are carried out.

Typically, and for illustration purposes only, a layer manure biomass material can have a total dry matter content (i.e. total solid content (TS)) of about 58% to about 66% (w/w), typically approx. 62% (w/w) and a content of volatile solids (VS) of about 50% (w/w) to about 58% (w/w), typically approx. 54% (w/w).

The below Table 17 illustrates the distribution of TAN (Total Ammonia N), uric acid N and protein N in a typical layer manure biomass

i) prior to pre-treatment in accordance with the methods of the present invention,

ii) after pre-treatment in accordance with the methods of the present invention, and prior to NiX treatment (i.e. thermo-chemical lime-pressure treatment), and

iii) after (NiX) nitrogen extraction, thermo-chemical lime-pressure cooking.

Analysis and determination of uric acid can be performed essentially according to Pekic et al.; Chromatographia; Vol. 27; No. 9/10; May 1989.

Analysis and determination of TAN can be performed essentially in accordance with the Kjeldahl analysis (Total-N) set out in ISO 5663 / DIN EN 25 663.

Initially, an untreated layer manure biomass may typically contain about 34 g organic N/kg TS - with an about equal distribution of the organic N pool between uric acid N and protein N.

The inorganic N pool typically amounts to less than about 15 g N/kg TS - depending on the specific biomass and the applied storage time and conditions. In the below example, the TAN contents are stated as 11.3 g N/kg TS.

Following a pre-treatment step in which the different organic N fractions of the layer manure biomass is subjected to a biological conversion resulting in a mineralization of the organic N fractions, essentially all of the uric acid and about half of the protein N is mineralized, thereby increasing the TAN pool to 36.7 g N/kg TS. TKN remains essentially unchanged as no ammonia N is yet stripped from the layer manure biomass.

Accordingly, by performing the biological pre-treatment step in accordance with the methods of the present invention, the availability of strippable TAN is increased from about 11.3 g N/kg TS to more than 3 times this amount - 36.7 g N/kg TS.

This is important as experiments have shown that the subsequent (NiX) thermo-chemical lime pressure cooking step is essentially unable to mineralize organic N, including protein N.

Hence, by increasing the availability of strippable, inorganic N (TAN), a significant reduction in TKN can be achieved – as evidenced by a TKN value of about 21.3 g N/kg TS for (NiX) thermo-chemical lime pressure cooked layer manure biomass. Note that protein N remains unchanged following the (NiX) thermo-chemical lime pressure cooking step.

Overall, the below-illustrated results demonstrate that it is possible, in accordance with the methods of the present invention, to achieve one or more of the following technical effects:

to convert essentially all of the uric acid present in the layer manure biomass to total ammonia N (TAN) during a biological, facultative anaerobic pre-treatment step as disclosed herein elsewhere in more detail;

to convert at least approximately 50%, such as for example at least approximately 55%, for example at least approximately 60%, such as for example at least approximately 65%, for example at least approximately 70%, such as for example at least approximately 75%, for example at least approximately 80%, of the organic N to inorganic N during the biological pre-treatment step as disclosed herein elsewhere in more detail;

to increase at least by a factor of approximately 3, such as at least by a factor of approximately 4, for example at least by a factor of approximately 5, as a result of the afore-mentioned organic material N conversions, the amount of total ammonia N (TAN) available for ammonia stripping during the (NiX) nitrogen extracting, thermo-chemical lime pressure cooking step as disclosed herein elsewhere in more detail;

to strip at least approximately 65%, such as at least approximately 70%, for example at least approximately 75%, such as at least approximately 80%, for example at least approximately 85%, of the total ammonia N TAN during the (NiX) nitrogen extracting, thermo-chemical lime pressure cooking step,

to reduce by at least approximately 50%, such as by at least approximately 55%, for example by at least approximately 60%, such as by at least approximately 65%, for example by at least approximately 70%; such as by at least approximately 75%, for example by at least approximately 80% - as a result of the stripping of total ammonia N (TAN) - the overall TKN value for the layer manure biomass in question, thereby significantly alleviating any inhibitory effect on the biogas formation exerted by the formation of ammonia in undesirable amounts during a subsequent biogas fermentation, i.e. amounts of ammonia which would have developed during the subsequent, anaerobic biogas fermentation in case the TKN value of the layer manure biomass had not been reduced as described herein elsewhere in more detail,

to convert the ratio of organic N : inorganic N from a ratio of more than 3 : 1 (gram organic N/kg TS : gram inorganic N/kg TS) prior to performing the biological, facultative anaerobic pre-treatment step of the methods of the present invention, to a ratio of at the most 1 : 1 (gram organic N/kg TS : gram inorganic N/kg TS), or less, such as a ratio of at the most 4 : 5 (gram organic N/kg TS : gram inorganic N/kg TS), for example a ratio of at the most 2 : 3 (gram organic N/kg TS : gram inorganic N/kg TS), such as a ratio of at the most 3 : 5 (gram organic N/kg TS : gram inorganic N/kg TS), for example a ratio of at the most 4 : 7 (gram organic N/kg TS : gram inorganic N/kg TS), prior to performing an anaerobic biogas fermentation, depending on the amount of organic N converted during the biological pre-treatment step and depending on the amount of TAN stripped during the (NiX) thermo-chemical lime pressure cooking step.

The figures presented in this example are illustrative only and should not be interpreted as constituting a limitation of the present invention.

The figures serve as an illustration of some of the important technical effects it is possible to achieve - under various practical circumstances - when exercising the present invention by subjecting e.g. typical layer manure biomass materials having a high content of organic N to the processing steps according to the present invention.

Untreated Layer Manure Biomass			**Mineralised Pre-treated Layer Manure Biomass**			**NiX-treated Layer Manure Biomass**		
	g/kg	g/kg TS		g/kg	g/kg TS		g/kg	g/kg TS
TAN	7.0	11.3	TAN	22.8	36.7	TAN	8.0	12.8
Uric acid	10.5	16.9	Uric acid	0.0	0.0	Uric acid	0.0	0.0
Protein	10.5	16.9	Protein	5.3	8.5	Protein	5.3	8.5
TKN	28.0	45.2	TKN	28.0	45.2	TKN	13.2	21.3

Table 17

Example 7

The below example further illustrates the technical effects achieved in accordance with the methods of the present invention.

The stated minimum and maximum values are representative minimum and maximum values of the contents of each of the N fractions presented in Table 18, i.e.:

TAN,
uric acid N; and
protein N.

Illustrative minimum and maximum values are stated for the different fractions for

a) an untreated, layer manure biomass (i.e. prior to pre-treatment and mineralization),

b) a layer manure biomass having been subjected to pre-treatment resulting in an N mineralization (i.e. after pre-treatment, but prior to NiX-treatment), and

c) a layer manure biomass having been subjected to NiX-treatment following pre-treatment.

	g/kg			g/kg TS		
Untreated	Min	Max	Layer manure	Min	Max	Layer manure
TAN	0.00	10.00	7.00	0.00	16.13	11.29
Uric acid N	0.00	15.00	10.50	0.00	24.19	16.94
Protein N	5.00	15.00	10.50	8.06	24.19	16.94
Mineralized	Min	Max	Layer manure	Min	Max	Layer manure
TAN	5.00	30.00	22.75	8.06	48.39	36.69
Uric acid N	0.00	10.00	0.00	0.00	16.13	0.00
Protein N	0.00	10.00	5.25	0.00	16.13	8.47
NiX-treated	Min	Max	Layer manure	Min	Max	Layer manure
TAN	0.00	15.00	7.96	0.00	24.19	12.84
Uric acid N	0.00	5.00	0.00	0.00	8.06	0.00
Protein N	0.00	10.00	5.25	0.00	16.13	8.47

Table 18

Claims

1. A method for generating biogas from an anaerobic fermentation of a processed, organic material comprising solid and liquid parts, said method comprising the steps of

   i) diverting a first organic material comprising one or more sources of nitrogen to a pre-incubation tank and subjecting said organic material to a mineralisation by chemical and/or biological means, wherein the mineralisation results in the conversion of organic N (nitrogen) present in the organic material to inorganic N;

   ii) diverting the pre-incubated first organic material to a lime pressure cooker and subjecting said first pre-incubated organic material to lime pressure cooking, wherein said lime pressure cooking step results in the formation of ammonia fluids, wherein said ammonia fluids are diverted from the lime pressure cooker and thereby separated from the organic material;

   iii) diverting pre-incubated and lime pressure cooked organic material from said lime pressure cooker to a buffer tank and lowering the pH value of the pre-incubated and lime pressure cooked material by contacting the pre-incubated and lime pressure cooked organic material in the buffer tank with a carbon dioxide (CO_2) containing gas.

2. The method of claim 1, wherein the mineralisation by chemical and/or biological means comprises a mineralisation by biological means and optionally also by chemical means.

3. The method of claim 2, wherein the mineralisation by biological means comprises the step of subjecting the organic material to a facultative, anaerobic fermentation, wherein organic N (nitrogen) present in the organic material is converted into inorganic N (nitrogen) during said anaerobic fermentation.

4. The method of claim 3, wherein the facultative, anaerobic fermentation essentially does not produce any biogas.

5. The method of claim 3, wherein said facultative, anaerobic fermentation is conducted at a temperature of from 15°C to preferably less than 42°C, such as a at temperature of from 20°C to preferably less than 40°C, for example at a temperature of from 25°C to

preferably less than 40°C, such as at a temperature of from about 30°C to about 40°C, for example at a temperature of about 37°C.

6. The method of claim 3, wherein the facultative, anaerobic fermentation is performed at a pH of from about 6.0 to a pH of from about 8.0, such as a pH of from about 6.0 to about 6.5, for example a pH of from about 6.5 to about 7.0, such as a pH of from about 7.0 to about 7.5, for example a pH of from about 7.5 to about 8.0.

7. The method of claim 3, wherein the facultative, anaerobic fermentation converts essentially all of the uric acid of the organic material into ammonium or ammonia comprising compounds.

8. The method of claim 3, wherein less than about 10% volatile solids, preferably less than about 5% volatile solids, more preferably less than about 2% volatile solids, and most preferably less than about 1% volatile solids are lost from the organic material during the facultative, anaerobic fermentation.

9. The method of claim 3, wherein the facultative, anaerobic fermentation is performed for a period of preferably less than about 96 hours, such as for a period of preferably less than 72 hours, for example for a period of preferably less than 48 hours, such as for a period of preferably less than 36 hours, for example for a period of preferably less than 24 hours.

10. The method of claim 3, wherein the facultative, anaerobic fermentation is performed by facultative anaerobic bacteria.

11. The method of claim 10, wherein the bacteria are facultative, anaerobic mesophilic bacteria.

12. The method of claim 3, wherein the facultative, anaerobic fermentation is carried out both in the absence as well as in the presence of atmospheric oxygen.

13. The method of claim 3, wherein the fermentation conditions are strictly anaerobic.

14. The method of claim 3, wherein the fermentation conditions are selected from aerobic fermentation conditions and anaerobic fermentation conditions, including a combination of both.

15. The method of claim 1, wherein the carbon dioxide (CO_2) containing gas is biogas.

16. The method of claim 1, wherein the carbon dioxide (CO_2) containing gas diverted to the organic material in the buffer tank is biogas diverted to the buffer tank from an anaerobic fermenter, or from a biogas storage facility connected thereto, wherein the anaerobic digester is connected to the buffer tank and receives input biomass material in the form of organic material present in the buffer tank.

17. The method of any of claims 1 and 16, wherein the diversion to the buffer tank of CO_2 containing gas, such as biogas, controls the pH of the organic material present in the buffer tank.

18. The method of claim 1, wherein at least approximately 50%, such as for example at least approximately 55%, for example at least approximately 60%, such as for example at least approximately 65%, for example at least approximately 70%, such as for example at least approximately 75%, for example at least approximately 80%, of the organic N is converted into inorganic N during the facultative, anaerobic fermentation.

19. The method of claim 1, wherein the amount of total ammonia N (TAN) available for ammonia stripping during the lime pressure cooking step is increased at least by a factor of approximately 3, such as at least by a factor of approximately 4, for example at least by a factor of approximately 5, as a result of the conversion of organic N into inorganic N during the facultative, anaerobic fermentation.

20. The method of claim 19, wherein at least approximately 65%, such as at least approximately 70%, for example at least approximately 75%, such as at least approximately 80%, for example at least approximately 85%, of the total ammonia N (TAN) is stripped during the lime pressure cooking step.

21. The method of claim 1, wherein the overall TKN value for the organic material is reduced by at least approximately 50%, such as by at least approximately 55%, for example by at least approximately 60%, such as by at least approximately 65%, for example by at least approximately 70%; such as by at least approximately 75%, for example by at least approximately 80% as a result of the stripping of total ammonia N (TAN) during the lime pressure cooking step.

22. The method of claim 21, wherein the reduction of TKN of the organic material significantly alleviates any undesirable and inhibitory effect on the biogas formation exerted by the formation of ammonia in undesirable amounts during a subsequent, anaerobic biogas fermentation.

23. The method of claim 1, wherein the ratio of organic N : inorganic N is converted from a ratio of more than 3 : 1 (gram organic N/kg TS : gram inorganic N/kg TS) prior to performing the facultative anaerobic fermentation, to a ratio of at the most 1 : 1 (gram organic N/kg TS : gram inorganic N/kg TS), or less, such as a ratio of at the most 4 : 5 (gram organic N/kg TS : gram inorganic N/kg TS), for example a ratio of at the most 2 : 3 (gram organic N/kg TS : gram inorganic N/kg TS), such as a ratio of at the most 3 : 5 (gram organic N/kg TS : gram inorganic N/kg TS), for example a ratio of at the most 4 : 7 (gram organic N/kg TS : gram inorganic N/kg TS), prior to performing the anaerobic biogas fermentation, as a result of i) the conversion of organic N to inorganic N which takes place during the facultative, anaerobic fermentation, and ii) the amount of ammonia N stripped during the lime pressure cooking step.

24. The method of claim 1, wherein the pre-incubated and lime pressure cooked organic material present in the buffer tank is diverted to an anaerobic biogas fermenter.

25. The method of claim 24, wherein the pH of the pre-incubated and lime pressure cooked organic material which is diverted from the buffer tank to the anaerobic biogas fermenter is in the range of from pH = 7.0 to 8.2.

26. The method of any of claims 24 and 25, wherein the pH value of the pre-incubated and lime pressure cooked organic material diverted to the anaerobic biogas fermenter is maintained in the anaerobic biogas fermenter within a predetermined pH-range by contacting the organic material present in the anaerobic biogas fermenter with re-circulated biogas, or a biogas diverted to the anaerobic biogas fermenter from an external source.

27. The method of claim 26, wherein the predetermined pH-range is preferably from pH = 7.0 to pH = 8.2.

28. The method of claim 1, wherein the ammonia fluids formed in the lime pressure cooker are diverted to an absorption unit, and absorbing and condensing the ammonia fluids diverted to the absorption unit from the lime pressure cooker.

29. The method of claim 1 comprising the further step of mixing in the buffer tank the lime pressure cooked organic material with a further organic material; wherein the further organic material preferably has not been subjected to lime pressure cooking prior to the mixing with the lime pressure cooked organic material.

30. The method of claim 29 comprising the further step of diverting the mixed, organic material(s) from the buffer tank to at least one, anaerobic biogas fermenter suitable for conducting an anaerobic, bacterial digestion of the organic material, wherein the anaerobic, bacterial digestion results in the generation of biogas.

31. The method of claim 1 comprising the further step of fermenting, under anaerobic fermentation conditions, the organic material diverted to the anaerobic biogas fermenter, and collecting the biogas resulting from said anaerobic fermentation of said organic material.

32. The method of claim 31 comprising the further step of diverting part or all of the organic material from the anaerobic biogas fermenter to a separation unit, and separating the organic material into a solid organic material fraction and a liquid organic material fraction comprising one or more sources of nitrogen selected from organic nitrogen sources and inorganic nitrogen sources.

33. The method of claim 32 comprising the further step of diverting said liquid organic material fraction comprising one or more sources of nitrogen to the pre-incubation tank and/or to the lime pressure cooker.

34. The method of claim 33 comprising the further step of mixing, in the pre-incubation tank and/or in the lime pressure cooker, said liquid fraction comprising one or more sources of nitrogen with a second organic material comprising one or more sources of nitrogen selected from organic nitrogen sources and inorganic nitrogen sources.

35. The method of claim 34 comprising the further step of mineralising the mixture of second organic material and liquid fraction by chemical and/or biological means, when the liquid fraction comprising one or more sources of nitrogen is diverted to the pre-incubation tank and mixed with the second organic material in the pre-incubation tank, wherein the mineralisation results in a conversion of organic N (nitrogen) present in the second organic material to inorganic N.

36. The method of any of claims 34 and 35 comprising the further step of stripping ammonia from the mixture of second organic material and liquid fraction, when the liquid fraction comprising one or more sources of nitrogen is diverted to the lime pressure cooker and mixed with the second organic material in the lime pressure cooker.

37. The method of claim 1, wherein lime comprises or essentially consists of CaO or $Ca(OH)_2$.

38. The method of claim 1, wherein the amount of added CaO used for lime pressure cooking is from about 2 to about 80 g per kg dry matter, such as from about 5 to about 60 g per kg dry matter.

39. The method of claim 1, wherein the lime pressure cooking of the organic material is performed at a temperature of from about 100°C to preferably less than 250°C, under a pressure of from about 2 to preferably less than 20 bar, such as a pressure of from about 2 to preferably less than 6 bar, and with addition of lime sufficient to reach a pH value of the organic material from about 9 to preferably less than 12.

40. The method of claim 1, wherein said organic material is fermented in a first, anaerobic fermenter under a first set of fermentation conditions, and subsequently diverted to a second, or further, anaerobic fermenter and fermented under a second or further set of fermentation conditions.

41. The method of claim 40, wherein said organic material is fermented under thermophile fermentation conditions and/or mesophile fermentation conditions.

42. The method of claim 41, wherein said organic material is initially fermented under thermophile fermentation conditions and subsequently under mesophile fermentation conditions.

43. The method of any of the claims 41 and 42, wherein biogas produced by thermophile and/or mesophile fermentation conditions is diverted to a gas storage facility operably connected to the one or more fermenters.

44. The method of any of claims 41 and 42, wherein the one or more biogas fermentation step(s) is/are performed at a temperature of from about 20°C to preferably less than about 65°C.

45. The method of any of claims 41 and 42, wherein the thermophilic reaction conditions include a reaction temperature ranging from 40°C to 65°C.

46. The method of any of claims 41 and 42, wherein the thermophilic reaction conditions include a reaction temperature ranging from 45°C to 60°C.

47. The method of any of claims 41 and 42, wherein the mesophilic reaction conditions include a reaction temperature ranging from 20°C to 40°C.

48. The method of any of claims 41 and 42, wherein the mesophilic reaction conditions include a reaction temperature ranging from 32°C to 38°C.

49. The method of any of the claims 41 and 42, wherein the thermophilic reaction is performed for about 5 to15 days, such as for about 7 to 10 days.

50. The method of any of the claims 41 and 42, wherein the mesophilic reaction is performed for about 5 to 15 days, such as for about 7 to 10 days.

51. A method for generating biogas from an anaerobic fermentation of processed organic material comprising solid and liquid parts, said method comprising the steps of

i) diverting a first organic material comprising one or more sources of nitrogen to a pre-incubation tank and subjecting said organic material to a mineralisation by chemical and/or biological means, wherein the mineralisation results in the conversion of organic N (nitrogen) present in the organic material to inorganic N;

ii) diverting the pre-incubated first organic material to a lime pressure cooker and subjecting said first pre-incubated organic material to lime pressure cooking, wherein said lime pressure cooking step results in the formation of ammonia fluids which are diverted from the lime pressure cooker and thereby separated from the first organic material;

iii) diverting pre-incubated and lime pressure cooked organic material from said lime pressure cooker to a buffer tank and lowering the pH value of the pre-incubated and lime pressure cooked material in the buffer tank.

52. The method of claim 51, wherein the pH value of the pre-incubated, organic material subjected to lime pressure cooking and ammonia stripping in the lime pressure cooker is preferably kept, during the lime pressure cooking treatment, within a pH range of from pH = 7.5 to pH = 12.0; for example from pH = 7.5 to pH = 11.5, such as from pH = 7.5 to pH = 11.0; for example from pH = 7.5 to pH = 10.5; such as from pH = 7.5 to pH = 10.0; for example from pH = 7.5 to pH = 9.5; such as from pH = 7.5 to pH = 9.0.

53. The method of any of claims 51, wherein the pH value of the pre-incubated and lime pressure cooked organic material in the buffer tank, when having been subjected to

fluids comprising CO_2 in amounts sufficient to reduce the pH value of the organic material, is preferably kept within a pH range of from pH = 7.0 to pH = 8.2; such as from pH = 7.0 to pH = 8.0; for example a pH of around 7.5.

54. The method of any of claims 51, wherein the pH value of the pre-incubated and lime pressure cooked organic material present in the buffer tank, after said organic material having been contacted with fluids comprising CO_2 in amounts sufficient to reduced the pH value of the organic material, is at least about 1 pH value lower than the pH value of the pre-incubated and lime pressure cooked organic material present in the lime pressure cooker.

55. The method of claim 51, wherein the lowering of the pH value of the pre-incubated and lime pressure cooked organic material present in the buffer tank is obtained i) by contacting said organic material in the buffer tank with a CO_2 containing gas, such as biogas, and optionally ii) by contacting said organic material in the buffer tank with an acid selected from an organic acid and an inorganic acid, and/or further optionally iii) by diverting, following anaerobic digestion and separation of the fermented, organic material, the separated liquid organic material fraction comprising one or more sources of nitrogen selected from organic nitrogen sources and inorganic nitrogen sources to the buffer tank.

56. The method of claim 51, wherein, during the pre-incubation step, if any biogas is produced, the amount of biogas constitutes less than 5 %, such as less than 4 %, for example less than 3 %, such as less than 2 %, for example less than 1 % of the amount of biogas produced by anaerobic biogas fermentation.

57. The method of claim 51, wherein percentage conversion of organic N into inorganic N in the per-incubation tank is around at least 35 % of the total organic fraction, such as at least 40%, preferably at least 45%, at least 50%, more preferably at least 55%, at least 60%, even more preferably at least 65%.

58. The method of claim 51, wherein the percentage conversion of organic N into inorganic N in the pre-incubation tank is around 70% to 80% of the total organic N fraction.

59. The method of claim 51, wherein, in the pre-incubation tank, nitrogen containing organic acids, such as uric acid, is converted to inorganic N in an amount of 80% or more of the organic acid fraction, such as 90% or more, preferably 95% or more, even more preferably around 100% of the organic acid fraction.

60. The method of claim 51, wherein, in the pre-incubation tank, organic bound nitrogen originating from protein is converted to inorganic N by a minimum of 30% of the organic bound N originating from protein fraction, such as by a minimum of 40%, by a minimum of 50%, by a minimum of 60% of the organic bound N originating from protein fraction.

61. The method of claim 51, wherein conversion of the organic N to inorganic N in the pre-incubation tank is performed at a temperature in the range of approximately 30 to 37°C, such as from 33 to 37°C, for example around 37°C.

62. The method of claim 51, wherein, when total solid (TS) content of the organic material in the lime pressure cooker is preferably less than approx. 30%, and preferably less than around 25%, such as less than around 23%, the rate of conversion of organic N to inorganic N in the pre-incubation tank is proportional to the amount of water present in the pre-incubation tank.

63. A dual fermentation method for generating biogas from an anaerobic fermentation of processed organic material comprising solid and liquid parts, said method comprising the steps of

i) subjecting an organic material comprising one or more sources of nitrogen to a facultative anaerobic fermentation resulting in at least partial hydrolysis of the organic material and at least partial conversion of organic N (nitrogen) present in the organic material to inorganic N, and wherein essentially no biogas is produced;

ii) subjecting the organic material fermented in step i) to lime pressure cooking, wherein the lime-pressure cooking results in a further hydrolysis of said organic material, and wherein said lime pressure cooking step results in the conversion of inorganic N to ammonia fluids; and

iii) subjecting the organic material fermented in step i) and subjected to lime pressure cooking in step ii) to a strictly anaerobic fermentation resulting in the production of biogas under conditions wherein the pH level of the anaerobic fermenter is kept within a predetermined pH range of from preferably pH = 7.0 to pH = 8.2 by contacting or injecting the organic material with fluids comprising CO_2 in an amount sufficient to achieve said pH control within said predetermined pH range.

64. The method of claim 63, wherein the pre-incubated and lime pressure cooked organic material is diverted from said lime pressure cooker to a buffer tank, and wherein the pH value of the pre-incubated and lime pressure cooked organic material in the buffer tank is lowered by contacting the pre-incubated and lime pressure cooked organic material in the buffer tank with an acid, such as an organic or inorganic acid, or by contacting the pre-incubated and lime pressure cooked organic material in the buffer tank with a carbon dioxide (CO_2) containing gas.

65. The method of claim 64, wherein the diversion to the buffer tank of CO_2 containing gas, or an organic or inorganic acid, controls the pH of the organic material diverted to the buffer tank and maintains said pH within an interval of from preferably pH = 7.0 to pH = 8.2.

66. The method of claim 65, wherein the pH of the organic material in the anaerobic fermenter is kept within a predetermined pH range of from preferably pH = 7.0 to pH = 8.2 by contacting or injecting the organic material in the anaerobic fermenter with fluids comprising CO_2 in an amount sufficient to achieve said pH control within said predetermined pH range.

67. The method of claim 63 to 66, wherein the facultative anaerobic fermentation converts at least about 75% (gram N / kg total dry matter), such as preferably at least about 80% (gram N / kg total dry matter) of the organic N selected from uric acid and protein N present in the organic material into inorganic N.

68. The method of claim 63 to 66, wherein the conversion of organic N (gram N / kg total dry matter) into inorganic N (gram N / kg total dry matter) during the facultative anaerobic fermentation increases at least by a factor of 3, such as preferably at least by a factor of 4, the amount of inorganic N (gram N / kg total dry matter) present in the organic material.

69. The method of claim 63 to 66, wherein at least about 65% (gram N / kg total dry matter), such as at least about 70% (gram N / kg total dry matter) of the inorganic N present in the organic material after the facultative anaerobic fermentation and prior to lime pressure cooking is stripped as gaseous ammonia during the lime pressure cooking step.

70. A method for generating biogas from an anaerobic fermentation of processed organic material comprising solid and liquid parts, said method comprising the steps of

a pre-incubation step comprising receiving in a pre-incubation tank an organic material having a first part removable inorganic N and organic N, and increasing the amount of the removable inorganic N by converting the organic N into a second part of the removable inorganic N using first fermentation conditions, wherein the pre-incubation step is free or at least substantially free from a generation of biogas;

a nitrogen stripping step comprising stripping the removable inorganic N, comprising the first part and the second part, from the pre-incubated organic material using a lime-pressure cooker for stripping said removable inorganic N; and

a second fermentation step comprising anaerobically fermenting, in an anaerobic biogas fermenter, the pre-incubated organic material that is mixed with a further organic material for the generation of biogas

wherein the organic material is contacted with a CO_2 containing gas either following the lime-pressure cooking step and prior to the anaerobic fermentation step, or during the anaerobic fermentation,

wherein the contacting of the organic material with the CO_2 containing gas results in lowering the pH of the organic material and in maintaining the pH of the organic material in the range of from preferably 7.0 to 8.2.

71. A plant for generating biogas from an anaerobic fermentation of processed organic material comprising solid and liquid parts, said plant comprising

i) a pre-incubation tank for mineralisation of an organic material by chemical or biological means; wherein the pre-incubation tank is operably connected to a lime pressure cooker; wherein the mineralisation results in the conversion of organic N (nitrogen) present in the organic material to inorganic N;

ii) a lime pressure cooker for further mineralisation of a first organic material comprising one or more sources of nitrogen;

iii) an absorption unit for absorbing and condensing ammonia fluids diverted to the absorption unit from the lime pressure cooker when said first organic material is subjected to lime pressure cooking;

iv) a buffer tank for mixing lime pressure cooked organic material with a further organic material prior to diverting the mixed, organic materials to one or more fermenters, said buffer tank being operably connected to a gas storage facility suitable for storage of CO_2 containing gas, such as biogas;

v) a gas storage facility suitable for storage of CO_2 containing gas, such as biogas, wherein the gas storage facility is operably connected to the buffer tank;

vi) one or more fermenters for anaerobically fermenting said organic materials diverted to said one or more fermenters from said buffer tank, wherein said fermentation results in the generation of biogas.

72. The plant according to claim 71, wherein the plant further comprises

vii) a separation unit for separating organic materials diverted to the separation unit from said one or more fermenters, wherein said separation of said organic materials results in the generation of a solid organic material fraction and a liquid fraction comprising one or more sources of nitrogen; and

viii) means for diverting said liquid fraction comprising one or more sources of nitrogen to the lime pressure cooker, wherein said liquid fraction is mixed in the lime pressure cooker with first organic material comprising one or more sources of nitrogen.

73. The plant according to any of claims 71 and 72, wherein the absorption unit for absorbing and condensing ammonia fluids diverted to the absorption unit from the lime pressure cooker comprises a steam condenser and a scrubber.

74. The plant according to any of claims 71 and 72, wherein the buffer tank is operably connected to a reception tank or a reception station suitable for receiving liquid and solid organic material, respectively.

75. The plant according to any of claims 71 and 72, wherein the buffer tank is further operably connected to the absorption unit for absorbing and condensing ammonia fluids, wherein said connection allows ammonia to be stripped in the buffer tank and diverted to the absorption unit.

76. The plant according to any of claims 71 and 72 comprising more than one fermenter for anaerobically fermenting said organic materials, wherein said more than one fermenters are serially connected so that organic material having been fermented in a first fermenter under a first set of fermentation conditions can be diverted to a second or further fermenter and fermented under a second or further set of fermentation conditions.

77. The plant according to any of claims 71 and 72, wherein said more than one fermenter comprises at least one primary fermenter suitable for thermophilic fermentation and at least one secondary fermenter suitable for mesophilic fermentation.

78. The plant according to any of claims 71 and 72 further comprising a biogas storage facility operably connected to the one or more fermenters.

Fig. 1

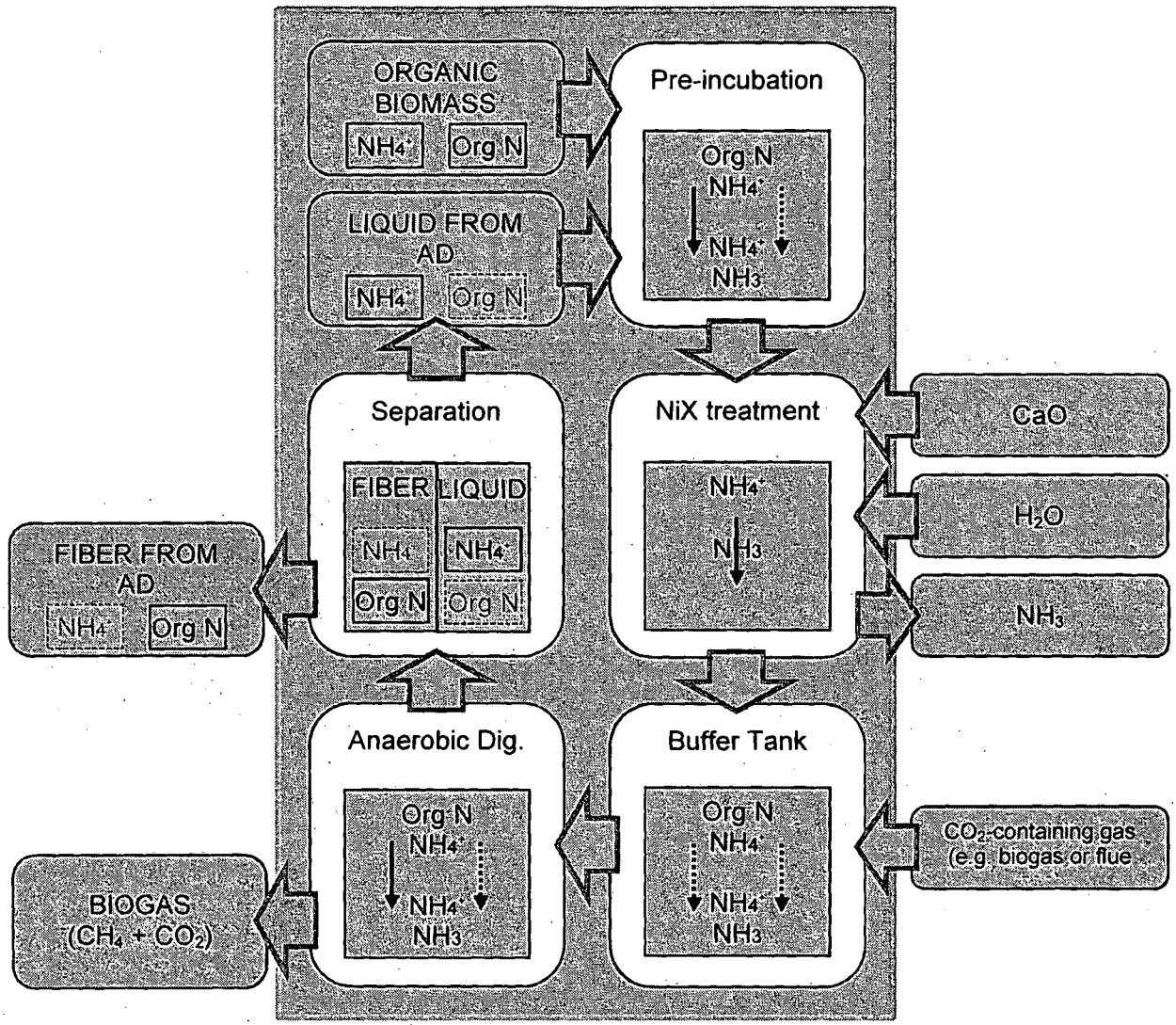

ORGANIC BIOMASS
NH4+ Org N
LIQUID FROM AD
NH4+ Org N
Pre-incubation
Org N
NH4+
NH4+
NH3
Separation
FIBER LIQUID
NH4+ NH4+
Org N Org N
NiX treatment
NH4+
NH3
CaO
H2O
NH3
FIBER FROM AD
NH4+ Org N
Anaerobic Dig.
Org N
NH4+
NH4+
NH3
Buffer Tank
Org N
NH4+
NH4+
NH3
CO2-containing gas (e.g. biogas or flue
BIOGAS (CH4 + CO2)

Fig. 2

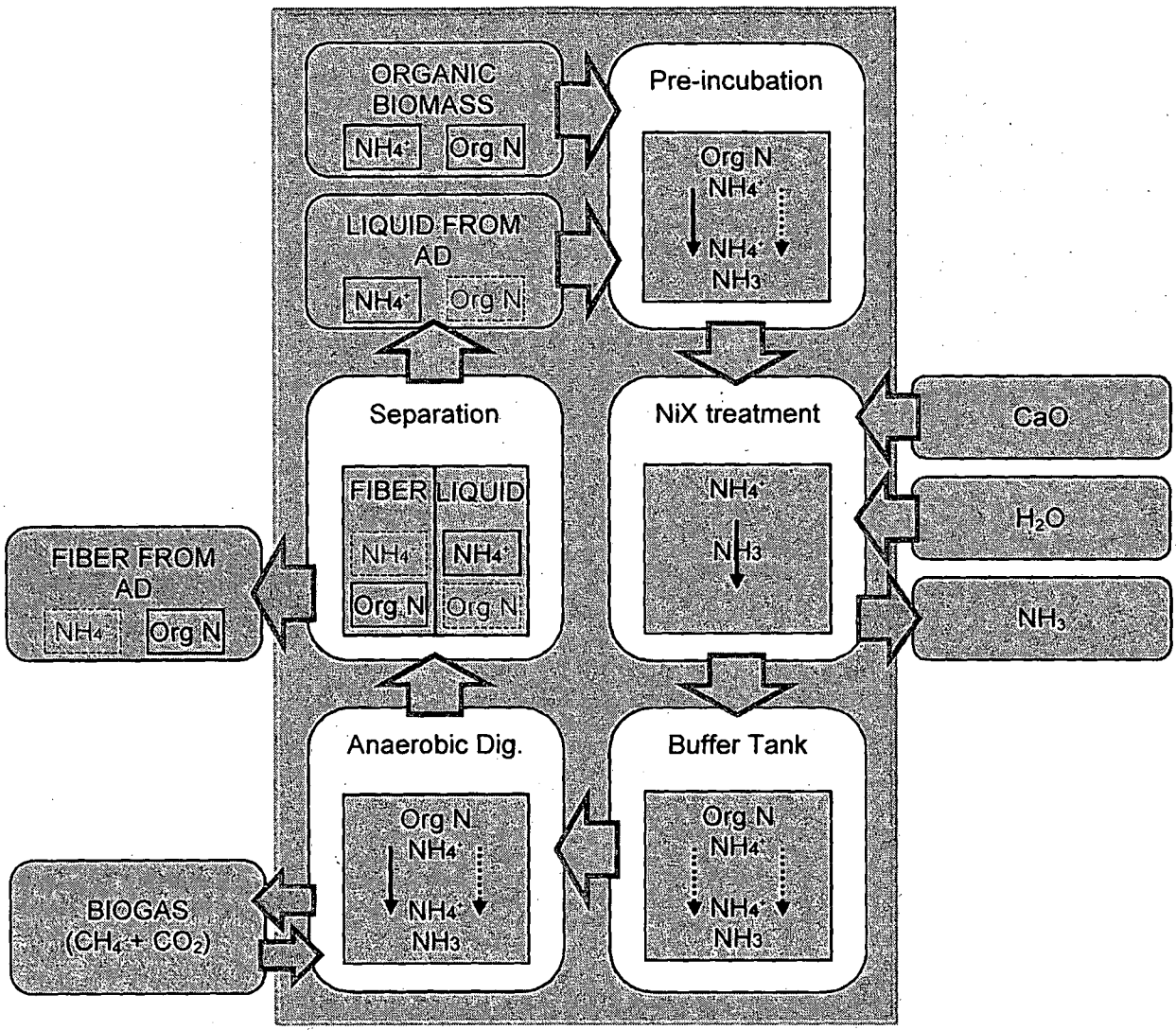

ORGANIC BIOMASS
NH4+ Org N
LIQUID FROM AD
NH4+ Org N
Pre-incubation
Org N
NH4+
NH4+
NH3
Separation
FIBER LIQUID
NH4+ NH4+
Org N Org N
NiX treatment
NH4+
NH3
CaO
H2O
NH3
FIBER FROM AD
NH4+ Org N
Anaerobic Dig.
Org N
NH4+
NH4+
NH3
Buffer Tank
Org N
NH4+
NH4+
NH3
BIOGAS
(CH4 + CO2)

Fig. 3

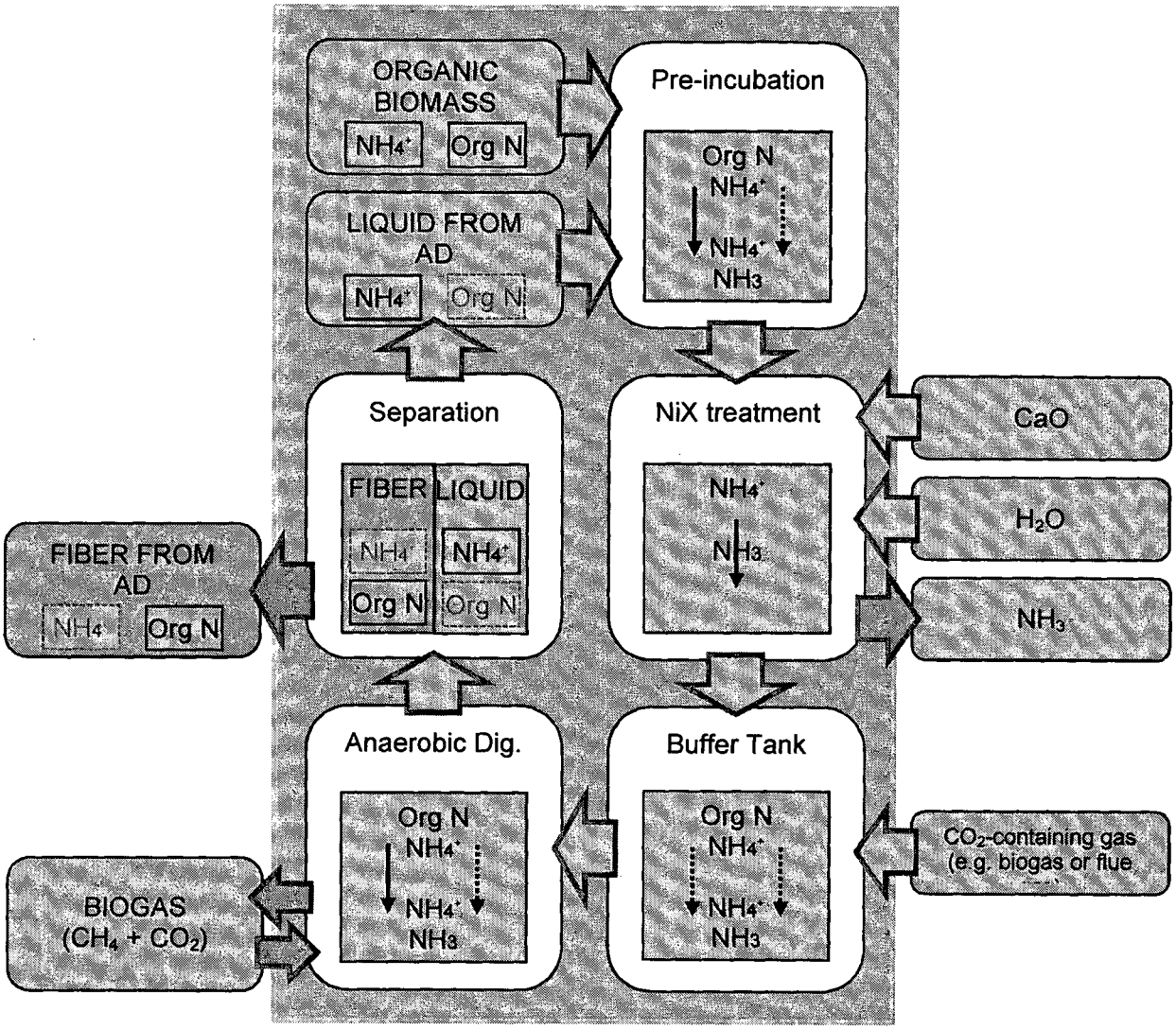

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LIQUID FROM AD
NH4+
Org N
Pre-incubation
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FIBER
LIQUID
NH4+
NH4+
Org N
Org N
NiX treatment
NH4+
NH3
CaO
H2O
NH3
FIBER FROM AD
NH4+
Org N
Anaerobic Dig.
Org N
NH4+
NH4+
NH3
Buffer Tank
Org N
NH4+
NH4+
NH3
CO2-containing gas (e.g. biogas or flue
BIOGAS (CH4 + CO2)

Fig. 4

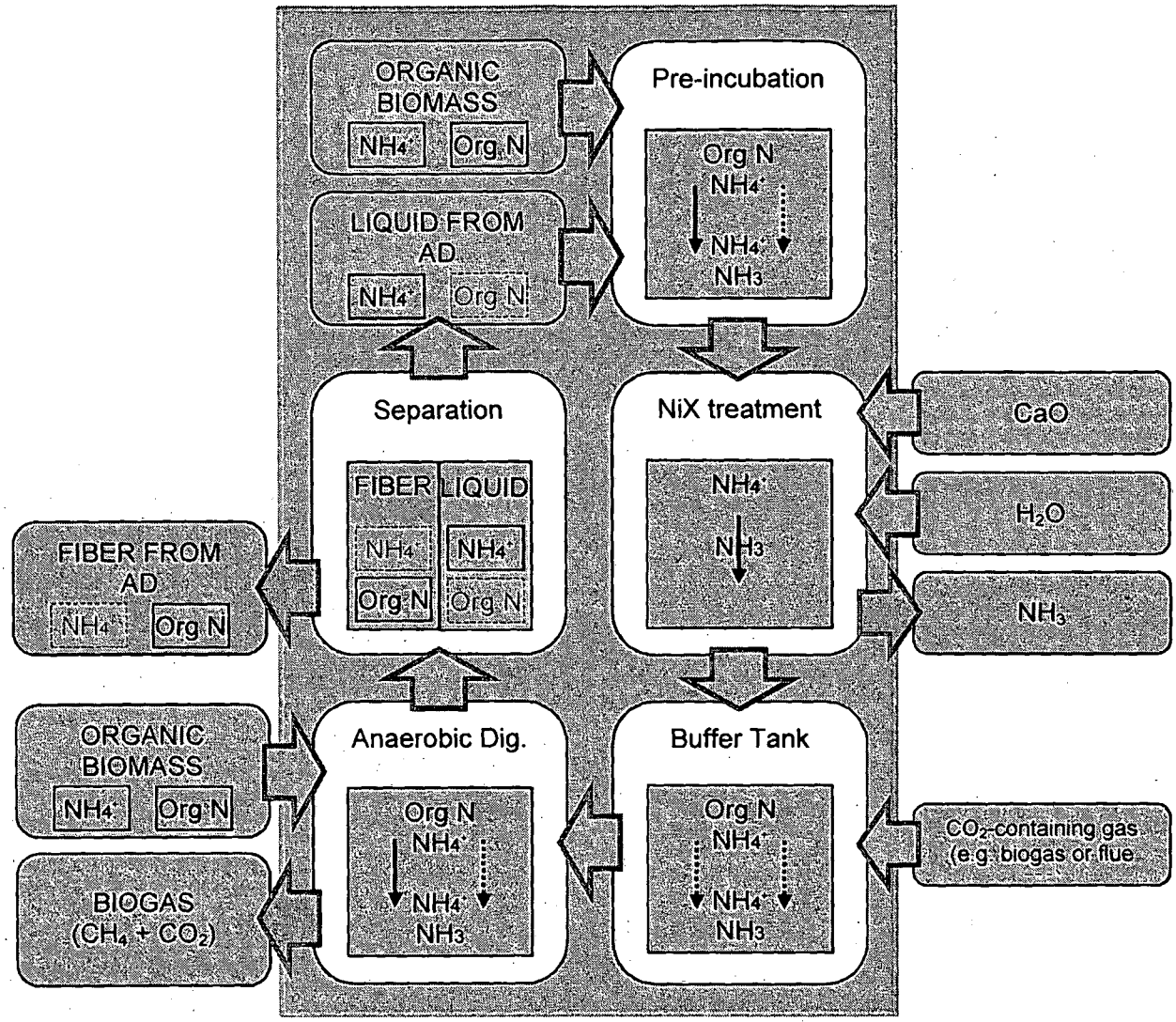

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LIQUID FROM AD
NH4+ Org N
Pre-incubation
Org N
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NH4+
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Separation
FIBER LIQUID
NH4+ NH4+
Org N Org N
NiX treatment
NH4+
NH3
CaO
H2O
NH3
FIBER FROM AD
NH4+ Org N
ORGANIC BIOMASS
NH4+ Org N
BIOGAS (CH4 + CO2)
Anaerobic Dig.
Org N
NH4+
NH4+
NH3
Buffer Tank
Org N
NH4+
NH4+
NH3
CO2-containing gas (e.g. biogas or flue

Fig. 5

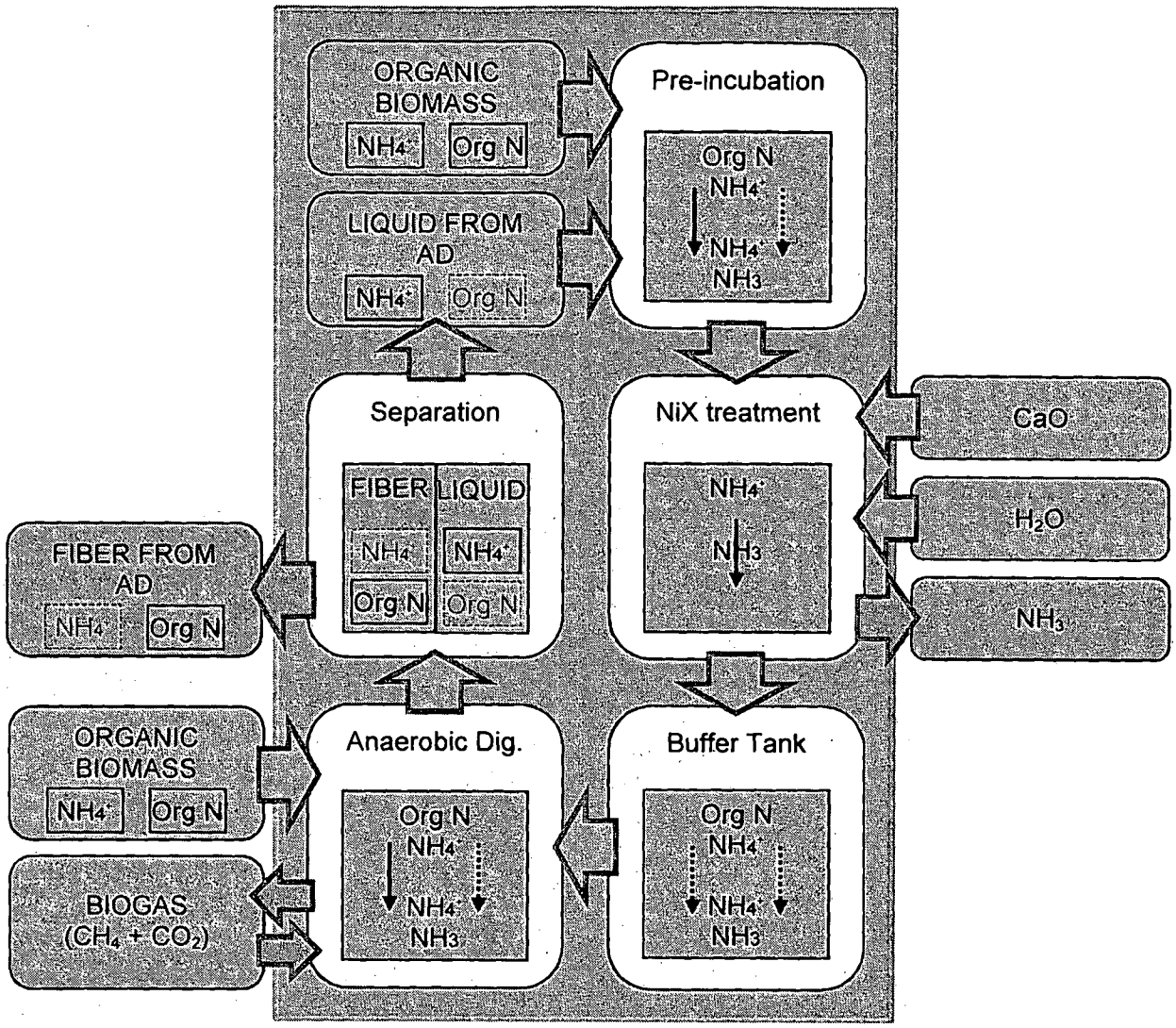

ORGANIC BIOMASS
NH4+ Org N
LIQUID FROM AD
NH4+ Org N
Pre-incubation
Org N
NH4+
NH4+
NH3
Separation
FIBER
LIQUID
NH4+
NH4+
Org N
Org N
NiX treatment
NH4+
NH3
CaO
H2O
NH3
FIBER FROM AD
NH4+ Org N
ORGANIC BIOMASS
NH4+ Org N
BIOGAS (CH4 + CO2)
Anaerobic Dig.
Org N
NH4+
NH4+
NH3
Buffer Tank
Org N
NH4+
NH4+
NH3

Fig. 6

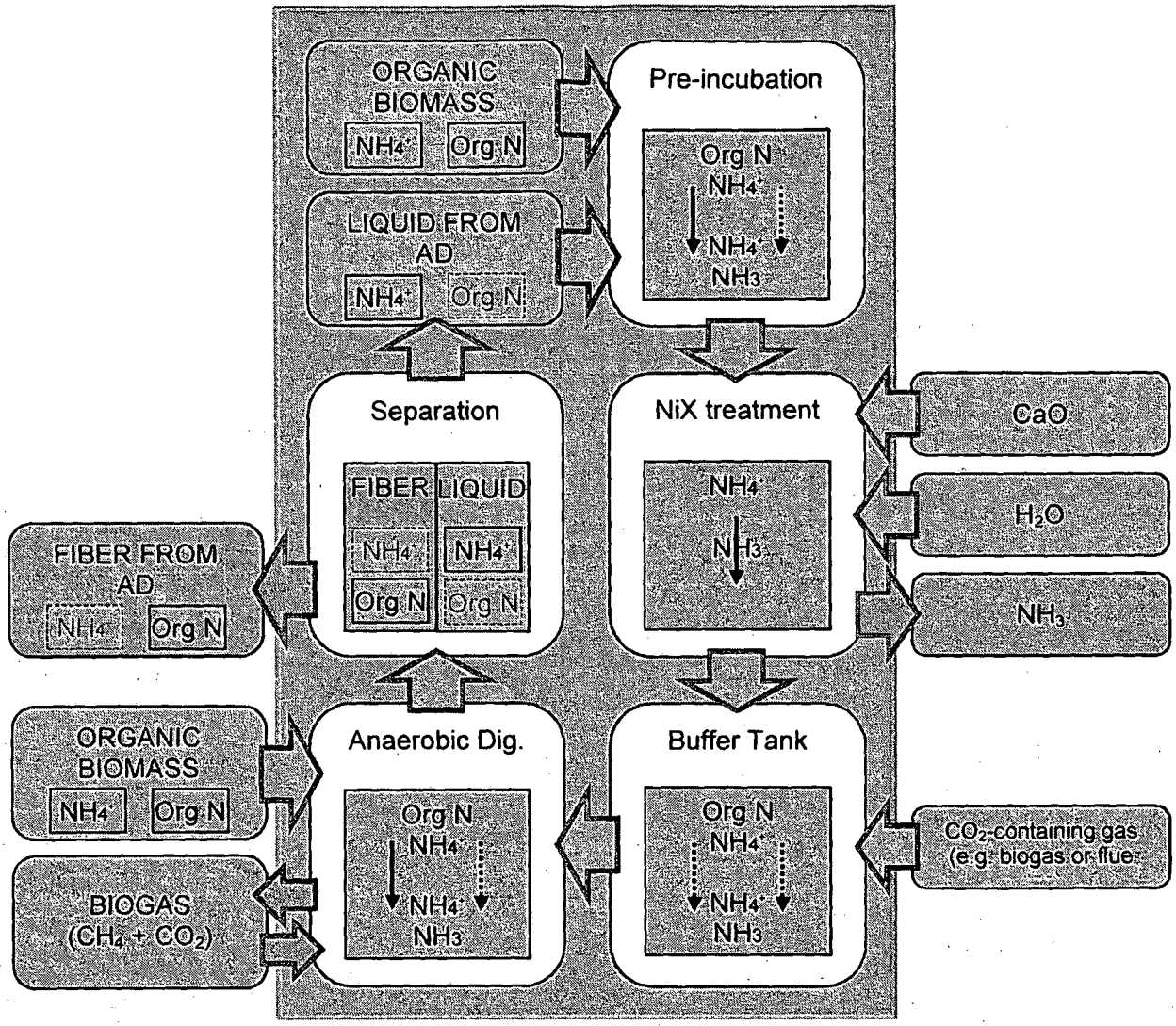

ORGANIC BIOMASS
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LIQUID FROM AD
NH4+ Org N
Pre-incubation
Org N
NH4+
NH4+
NH3
Separation
FIBER LIQUID
NH4+ NH4+
Org N Org N
NiX treatment
NH4+
NH3
CaO
H2O
NH3
FIBER FROM AD
NH4+ Org N
ORGANIC BIOMASS
NH4+ Org N
BIOGAS (CH4 + CO2)
Anaerobic Dig.
Org N
NH4+
NH4+
NH3
Buffer Tank
Org N
NH4+
NH4+
NH3
CO2-containing gas (e.g. biogas or flue

Fig. 7

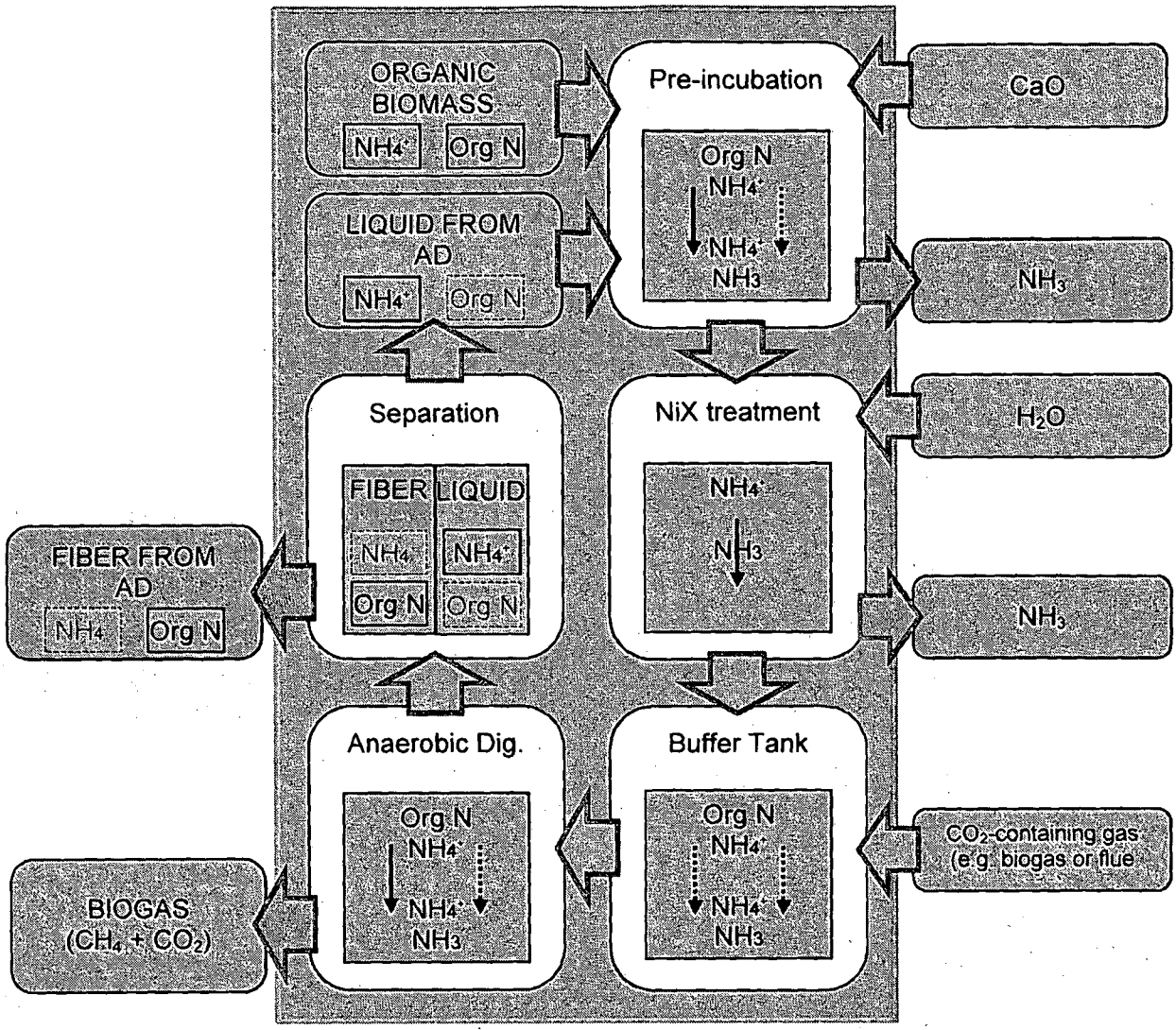

ORGANIC BIOMASS
NH4+
Org N
LIQUID FROM AD
NH4+
Org N
Pre-incubation
Org N
NH4+
NH4+
NH3
CaO
NH3
Separation
FIBER
LIQUID
NH4+
NH4+
Org N
Org N
NiX treatment
NH4+
NH3
H2O
NH3
FIBER FROM AD
NH4+
Org N
Anaerobic Dig.
Org N
NH4+
NH4+
NH3
Buffer Tank
Org N
NH4+
NH4+
NH3
CO2-containing gas (e.g. biogas or flue
BIOGAS (CH4 + CO2)

Fig. 8

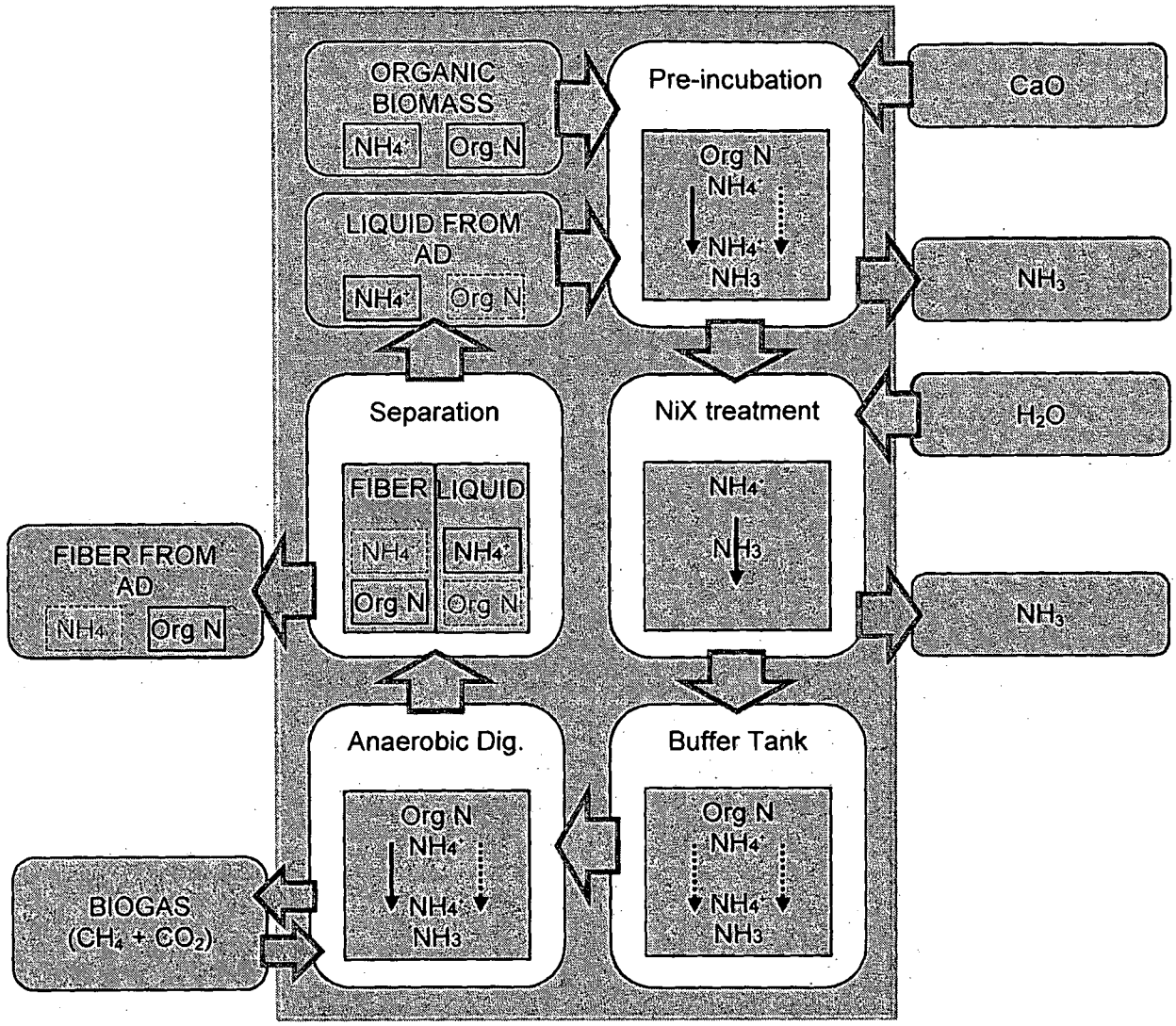

ORGANIC BIOMASS
NH4+ Org N
LIQUID FROM AD
NH4+ Org N
Pre-incubation
Org N
NH4+
NH4+
NH3
CaO
NH3
Separation
FIBER LIQUID
NH4+ NH4+
Org N Org N
NiX treatment
NH4+
NH3
H2O
NH3
FIBER FROM AD
NH4+ Org N
Anaerobic Dig.
Org N
NH4+
NH4+
NH3
Buffer Tank
Org N
NH4+
NH4+
NH3
BIOGAS
(CH4 + CO2)

Fig. 9

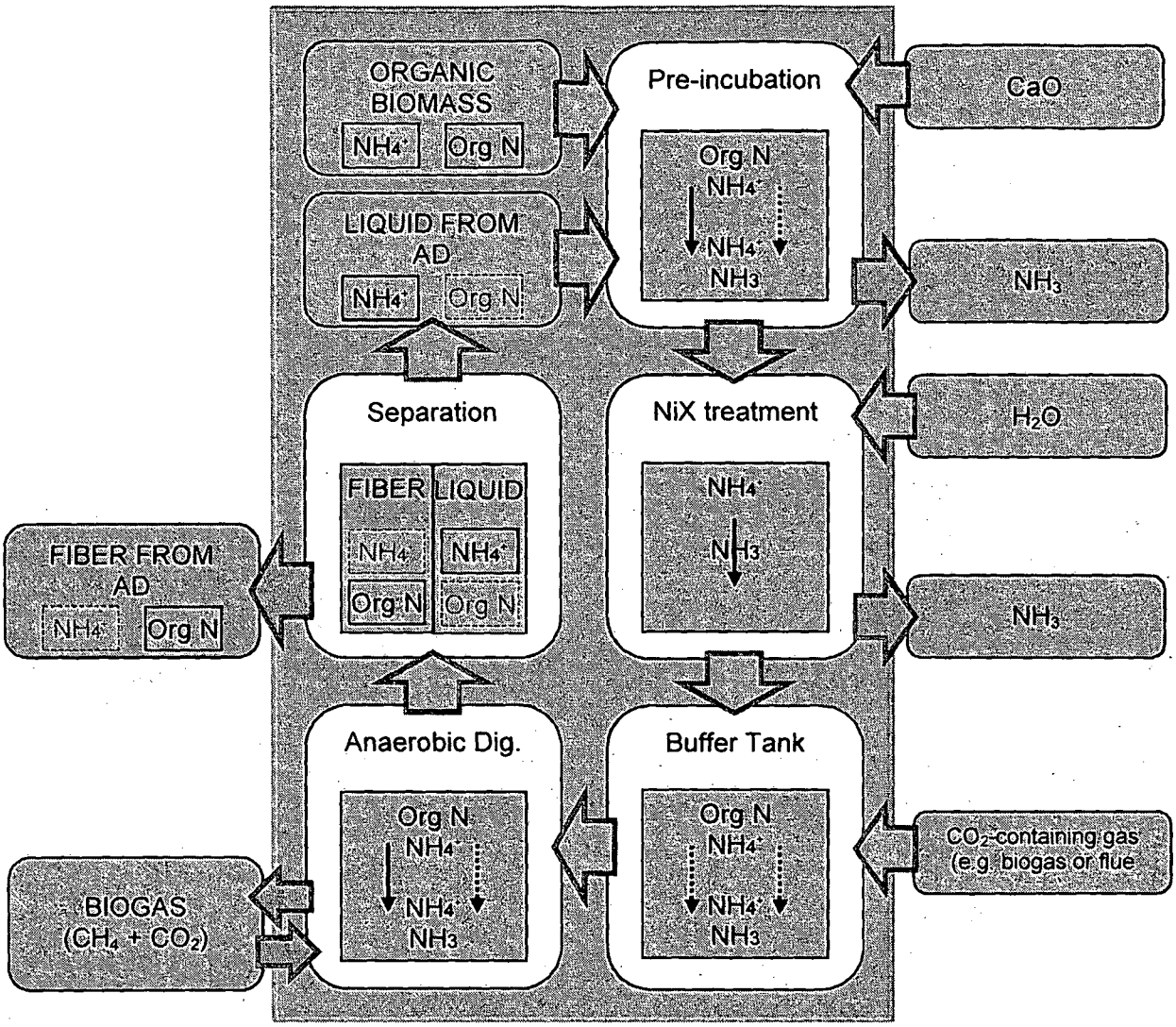

ORGANIC BIOMASS
NH4+ Org N
LIQUID FROM AD
NH4+ Org N
FIBER FROM AD
NH4+ Org N
BIOGAS (CH4 + CO2)
Pre-incubation
Org N
NH4+
NH4+
NH3
Separation
FIBER LIQUID
NH4+ NH4+
Org N Org N
NiX treatment
NH4+
NH3
Anaerobic Dig.
Org N
NH4+
NH4+
NH3
Buffer Tank
Org N
NH4+
NH4+
NH3
CaO
NH3
H2O
NH3
CO2-containing gas (e.g. biogas or flue

Fig. 10

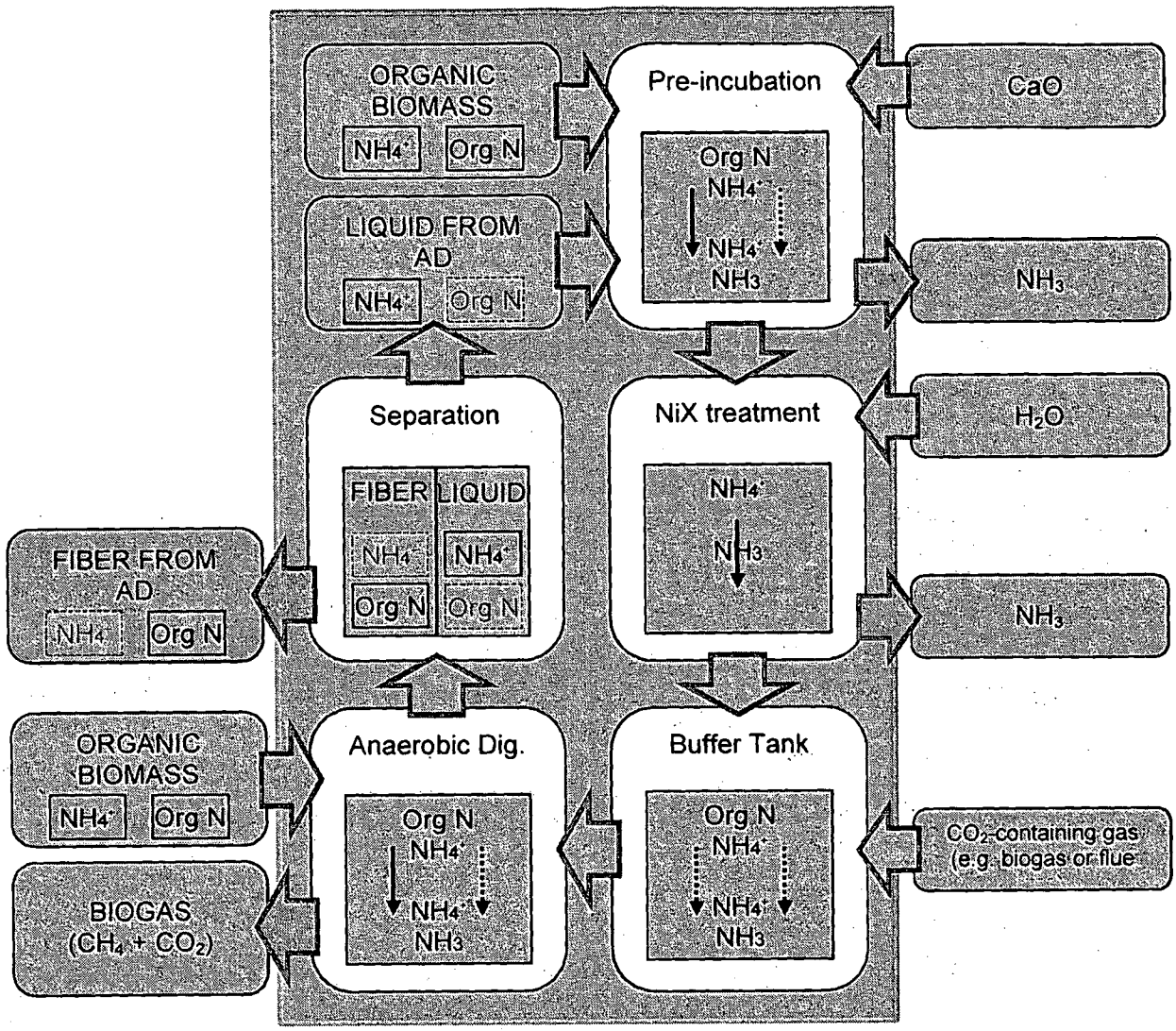

ORGANIC BIOMASS
NH4+ Org N
LIQUID FROM AD
NH4+ Org N
FIBER FROM AD
NH4+ Org N
ORGANIC BIOMASS
NH4+ Org N
BIOGAS (CH4 + CO2)
Pre-incubation
Org N
NH4+
NH4+
NH3
Separation
FIBER LIQUID
NH4+ NH4+
Org N Org N
NiX treatment
NH4+
NH3
Anaerobic Dig.
Org N
NH4+
NH4+
NH3
Buffer Tank
Org N
NH4+
NH4+
NH3
CaO
NH3
H2O
NH3
CO2-containing gas (e.g. biogas or flue

Fig. 11

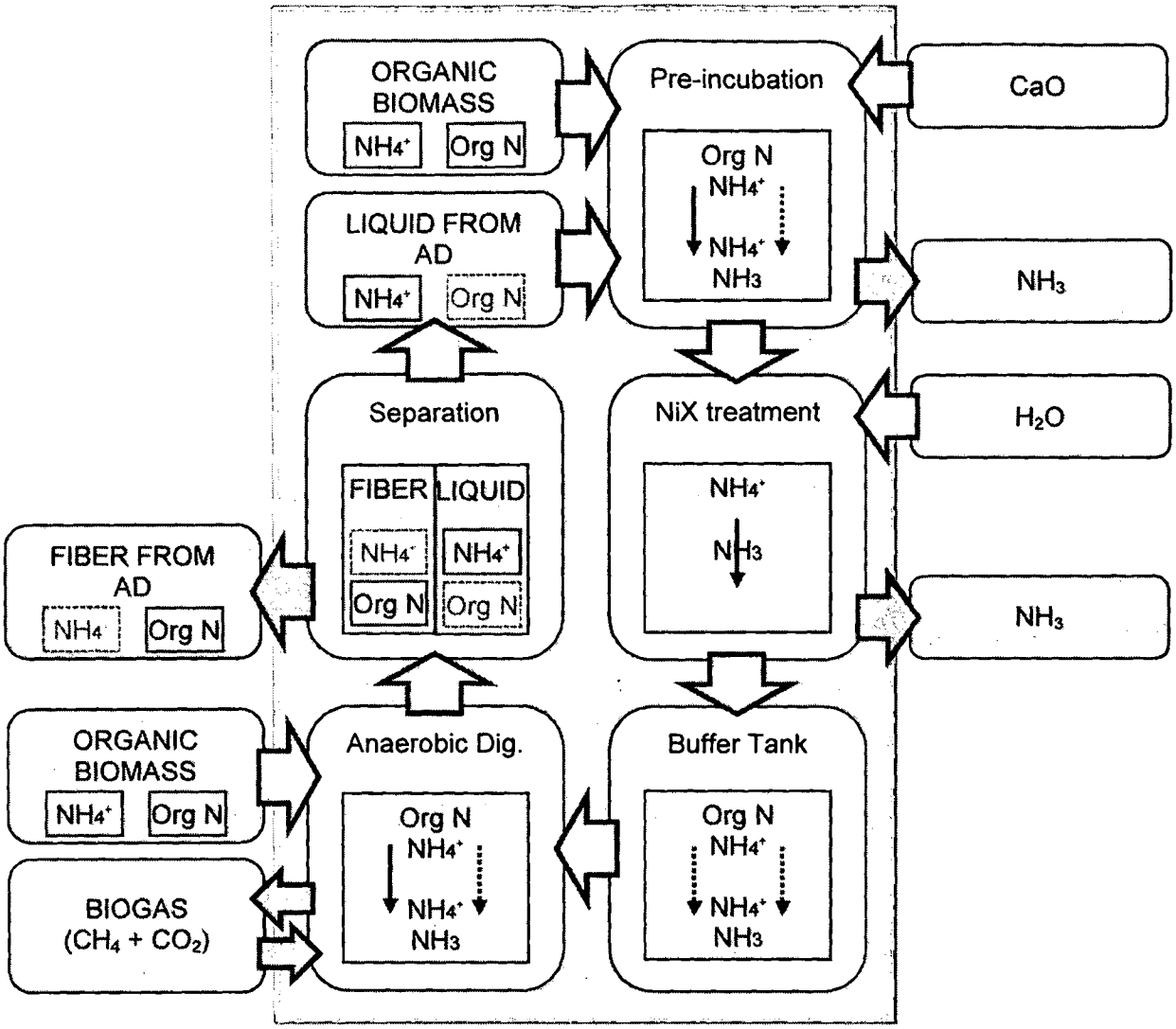

ORGANIC BIOMASS
NH4+ Org N
LIQUID FROM AD
NH4+ Org N
Pre-incubation
Org N
NH4+
NH4+
NH3
CaO
NH3
Separation
FIBER LIQUID
NH4+ NH4+
Org N Org N
NiX treatment
NH4+
NH3
H2O
NH3
FIBER FROM AD
NH4+ Org N
ORGANIC BIOMASS
NH4+ Org N
BIOGAS (CH4 + CO2)
Anaerobic Dig.
Org N
NH4+
NH4+
NH3
Buffer Tank
Org N
NH4+
NH4+
NH3

Fig. 12

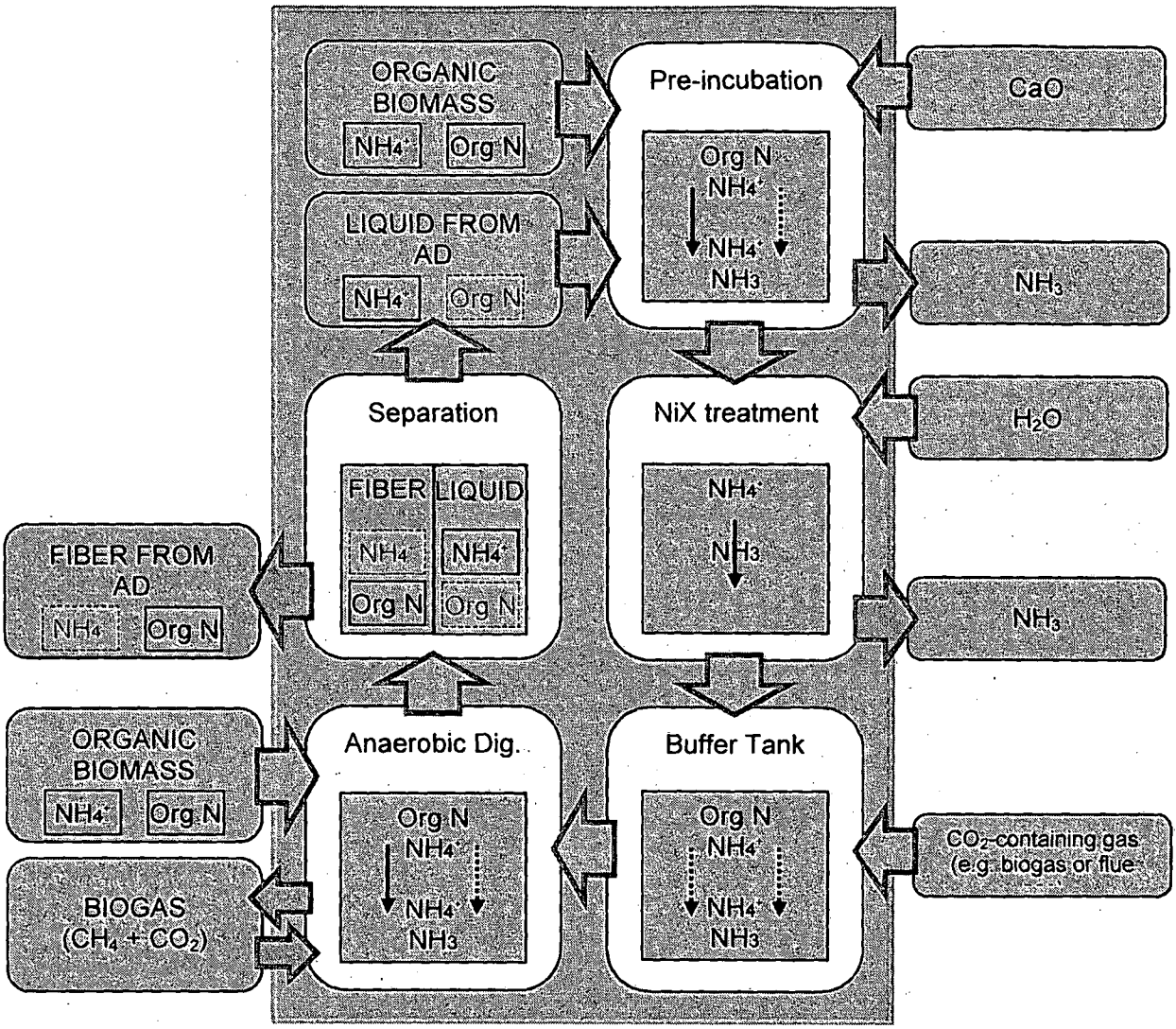

ORGANIC BIOMASS
NH4+ Org N
LIQUID FROM AD
NH4+ Org N
FIBER FROM AD
NH4+ Org N
ORGANIC BIOMASS
NH4+ Org N
BIOGAS (CH4 + CO2)
Pre-incubation
Org N
NH4+
NH4+
NH3
Separation
FIBER LIQUID
NH4+ NH4+
Org N Org N
NiX treatment
NH4+
NH3
Anaerobic Dig.
Org N
NH4+
NH4+
NH3
Buffer Tank
Org N
NH4+
NH4+
NH3
CaO
NH3
H2O
NH3
CO2-containing gas (e.g. biogas or flue

Fig. 13

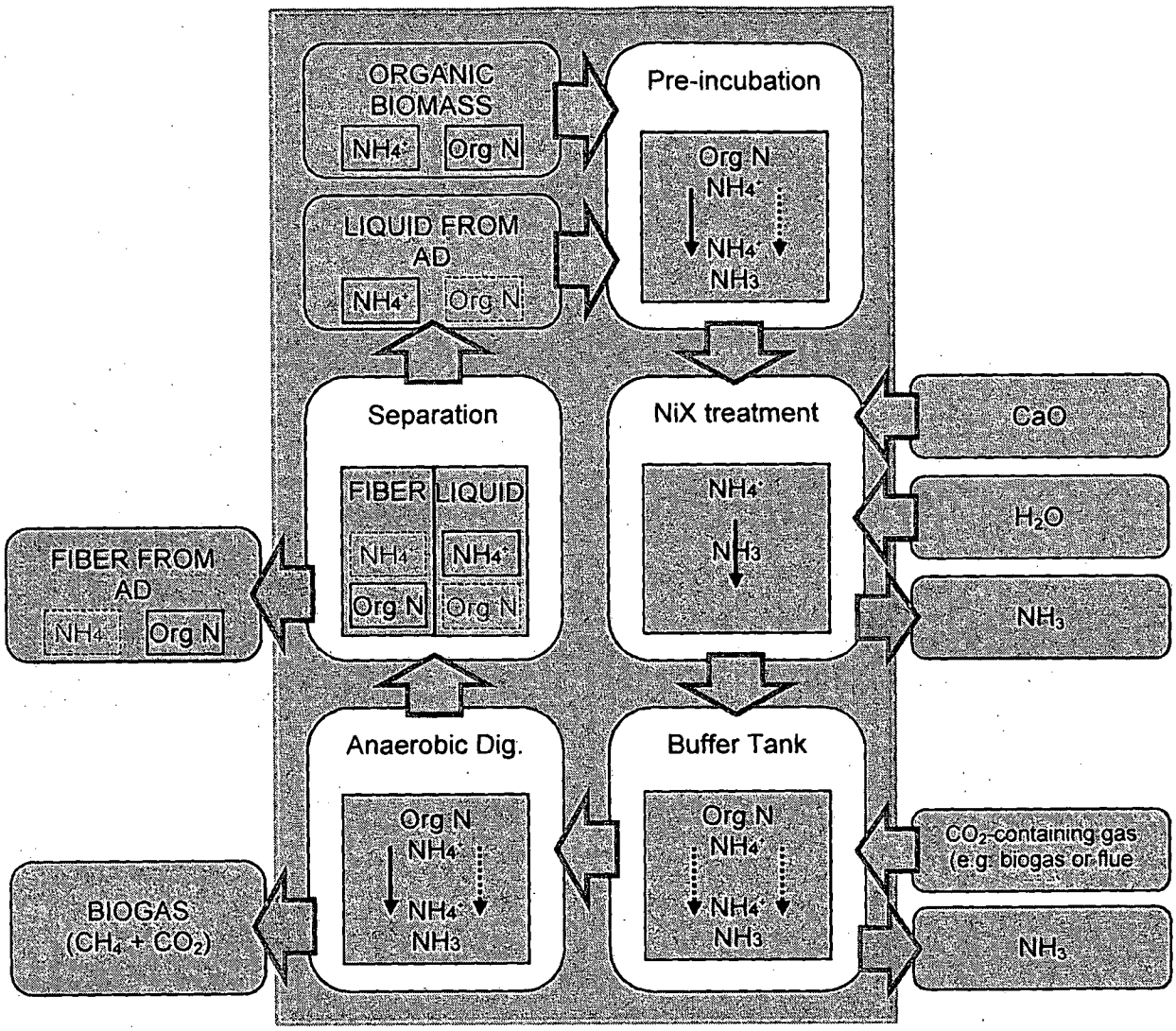

ORGANIC BIOMASS
NH4+ Org N
LIQUID FROM AD
NH4+ Org N
Pre-incubation
Org N
NH4+
NH4+
NH3
Separation
FIBER LIQUID
NH4+ NH4+
Org N Org N
NiX treatment
NH4+
NH3
CaO
H2O
NH3
FIBER FROM AD
NH4+ Org N
Anaerobic Dig.
Org N
NH4+
NH4+
NH3
Buffer Tank
Org N
NH4+
NH4+
NH3
BIOGAS (CH4 + CO2)
CO2-containing gas (e.g. biogas or flue
NH3

Fig. 14

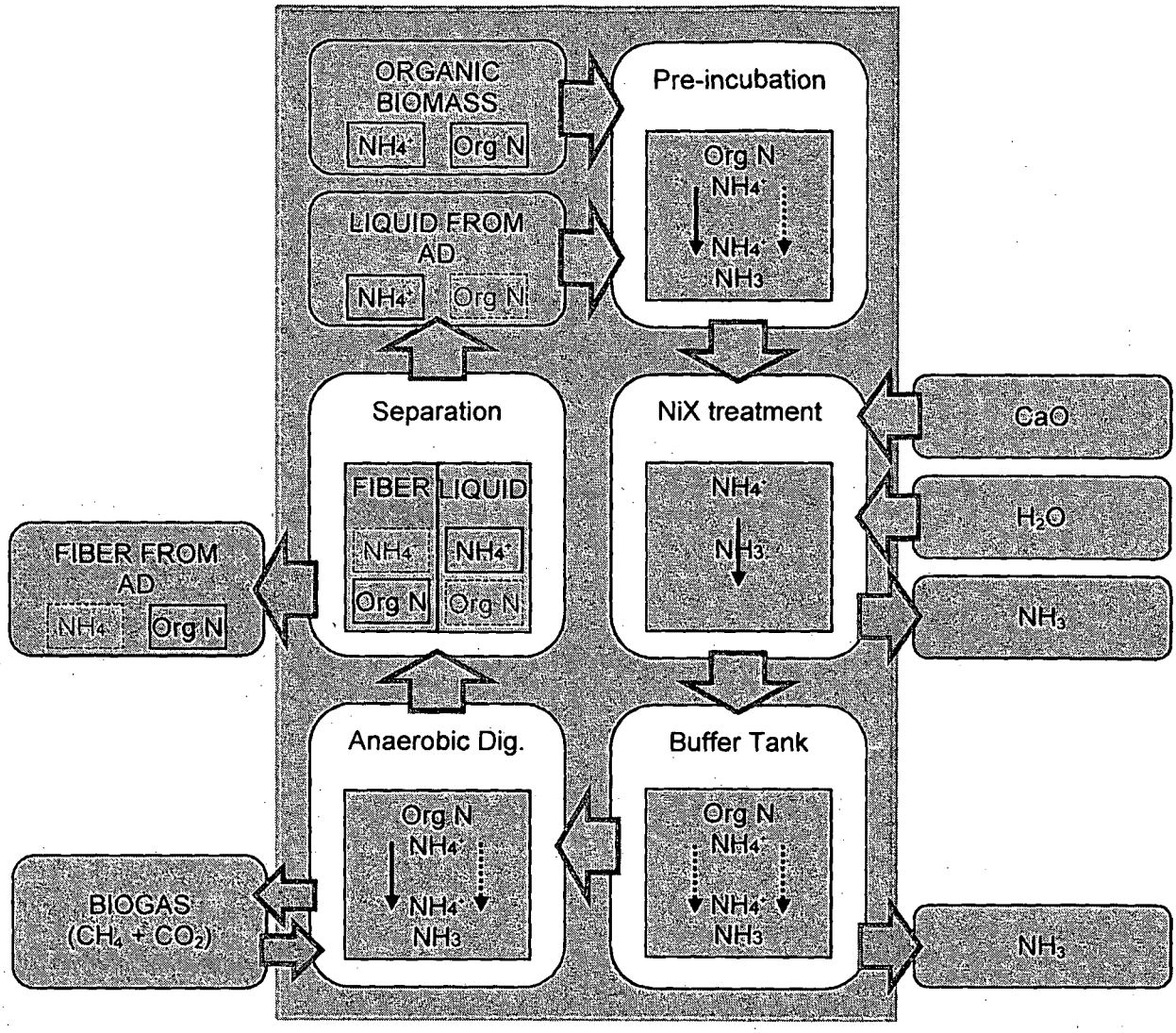

ORGANIC BIOMASS
NH4+ Org N
LIQUID FROM AD
NH4+ Org N
Pre-incubation
Org N
NH4+
NH4+
NH3
Separation
FIBER LIQUID
NH4+ NH4+
Org N Org N
NiX treatment
NH4+
NH3
CaO
H2O
NH3
FIBER FROM AD
NH4+ Org N
Anaerobic Dig.
Org N
NH4+
NH4+
NH3
Buffer Tank
Org N
NH4+
NH4+
NH3
BIOGAS (CH4 + CO2)
NH3

Fig. 15

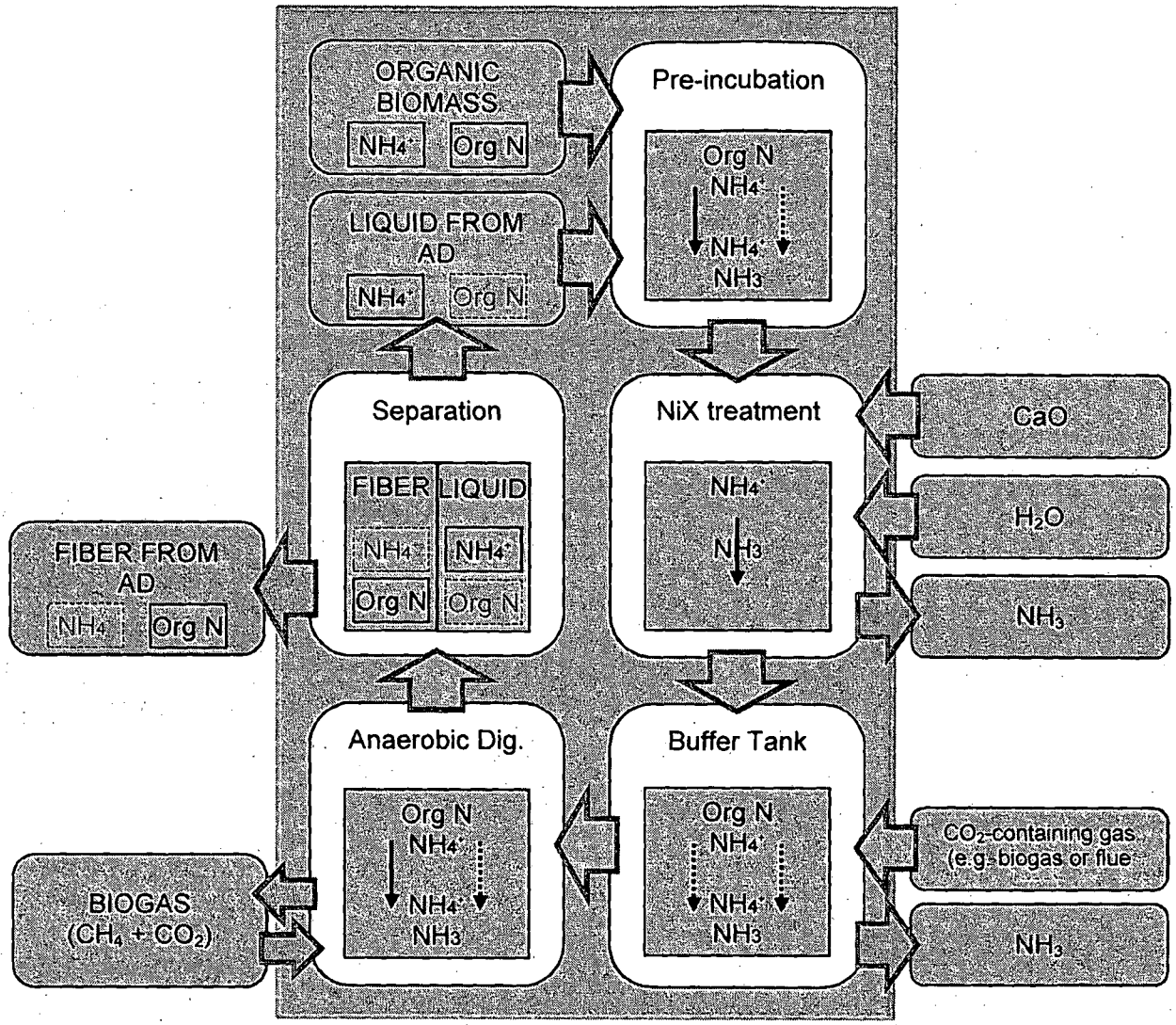

ORGANIC BIOMASS
NH4+ Org N
LIQUID FROM AD
NH4+ Org N
Pre-incubation
Org N
NH4+
NH4+
NH3
Separation
FIBER LIQUID
NH4+ NH4+
Org N Org N
NiX treatment
NH4+
NH3
CaO
H2O
NH3
FIBER FROM AD
NH4+ Org N
Anaerobic Dig.
Org N
NH4+
NH4+
NH3
Buffer Tank
Org N
NH4+
NH4+
NH3
BIOGAS (CH4 + CO2)
CO2-containing gas (e.g. biogas or flue
NH3

Fig. 16

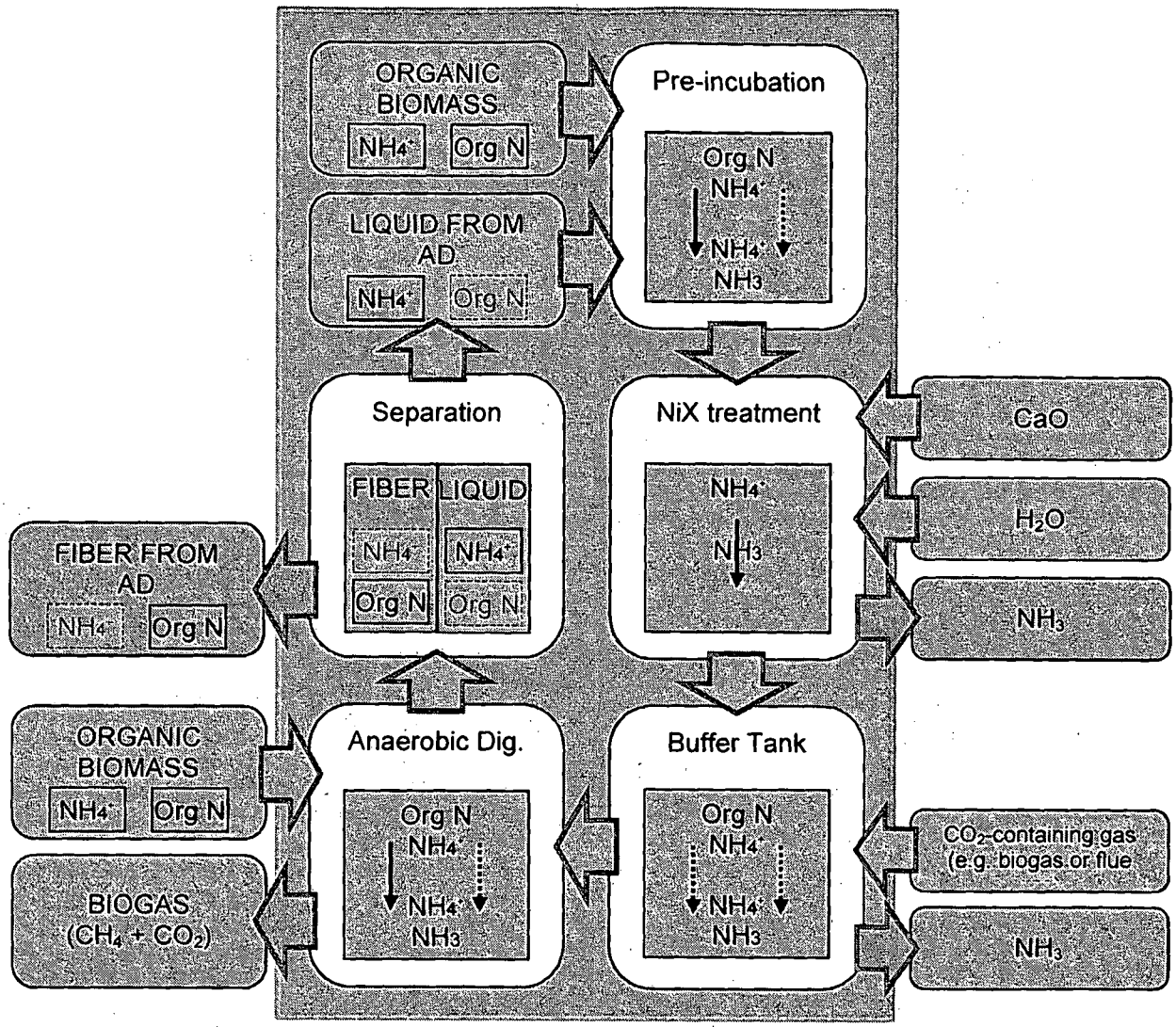

ORGANIC BIOMASS
NH4 Org N
LIQUID FROM AD
NH4 Org N
Pre-incubation
Org N
NH4
NH4
NH3
Separation
FIBER LIQUID
NH4 NH4
Org N Org N
NiX treatment
NH4
NH3
CaO
H2O
NH3
FIBER FROM AD
NH4 Org N
ORGANIC BIOMASS
NH4 Org N
BIOGAS (CH4 + CO2)
Anaerobic Dig.
Org N
NH4
NH4
NH3
Buffer Tank
Org N
NH4
NH4
NH3
CO2-containing gas (e.g. biogas or flue
NH3

Fig. 17

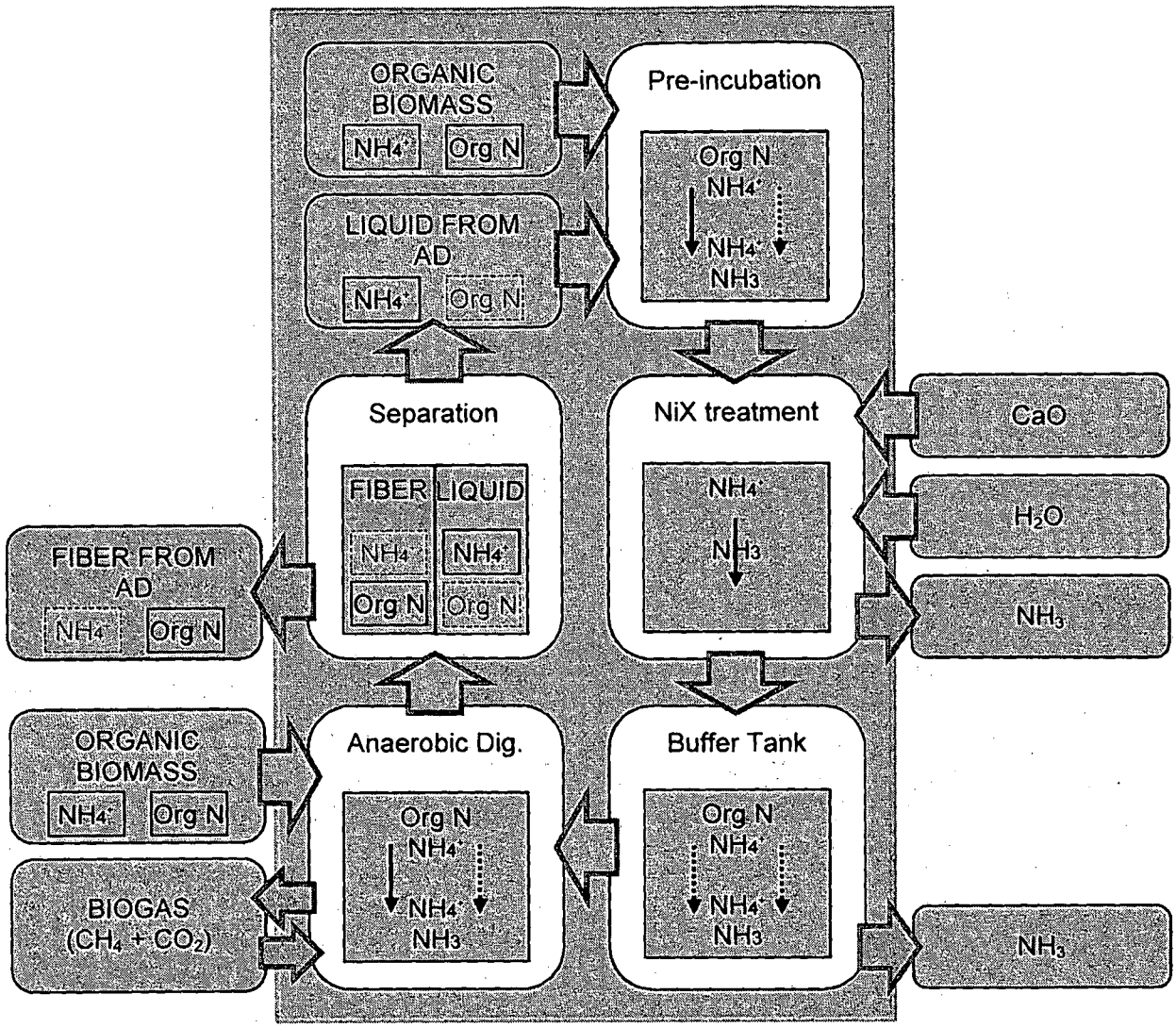

ORGANIC BIOMASS
NH4+
Org N
LIQUID FROM AD
NH4+
Org N
Pre-incubation
Org N
NH4+
NH4+
NH3
Separation
FIBER
LIQUID
NH4+
NH4+
Org N
Org N
NiX treatment
NH4+
NH3
CaO
H2O
NH3
FIBER FROM AD
NH4+
Org N
ORGANIC BIOMASS
NH4+
Org N
BIOGAS (CH4 + CO2)
Anaerobic Dig.
Org N
NH4+
NH4+
NH3
Buffer Tank
Org N
NH4+
NH4+
NH3
NH3

Fig. 18

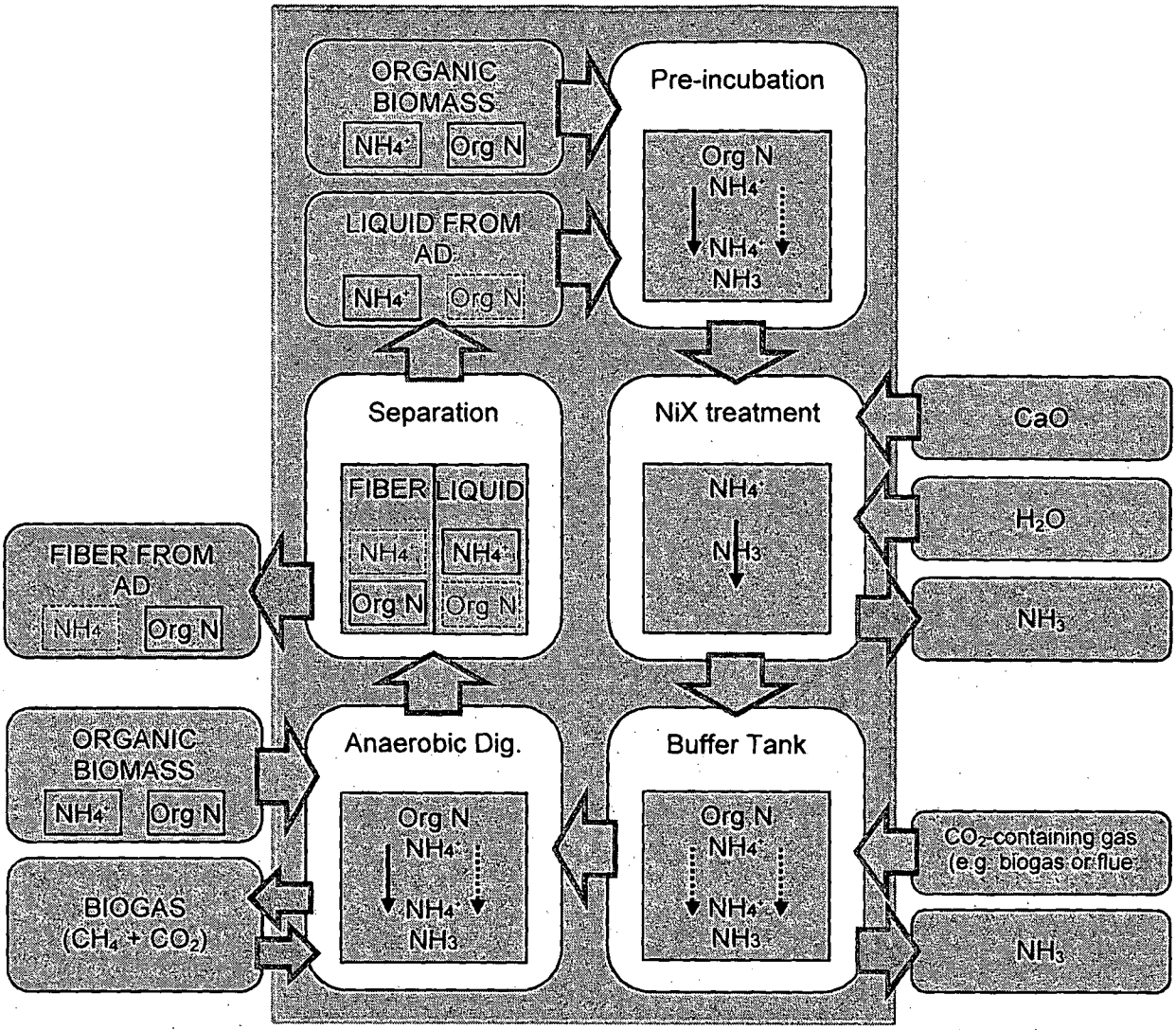

ORGANIC BIOMASS
NH4+ Org N
LIQUID FROM AD
NH4+ Org N
Pre-incubation
Org N
NH4+
NH4+
NH3
Separation
FIBER LIQUID
NH4+ NH4+
Org N Org N
NiX treatment
NH4+
NH3
CaO
H2O
NH3
FIBER FROM AD
NH4+ Org N
ORGANIC BIOMASS
NH4+ Org N
BIOGAS (CH4 + CO2)
Anaerobic Dig.
Org N
NH4+
NH4+
NH3
Buffer Tank
Org N
NH4+
NH4+
NH3
CO2-containing gas (e.g. biogas or flue)
NH3

Fig. 19

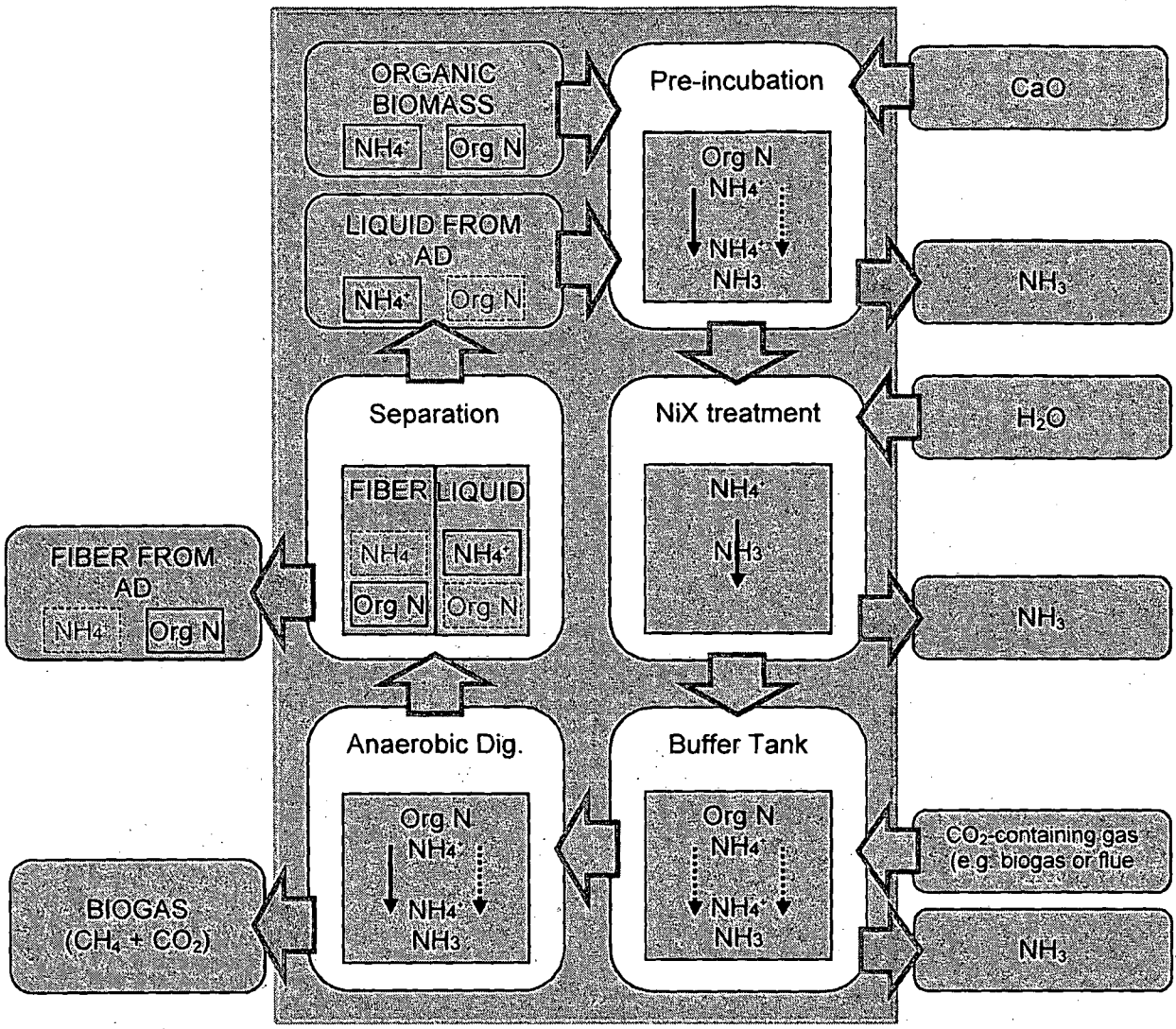

ORGANIC BIOMASS
NH4
Org N
LIQUID FROM AD
NH4
Org N
Pre-incubation
Org N
NH4
NH4
NH3
CaO
NH3
Separation
FIBER
LIQUID
NH4
NH4
Org N
Org N
NiX treatment
NH4
NH3
H2O
NH3
FIBER FROM AD
NH4
Org N
Anaerobic Dig.
Org N
NH4
NH4
NH3
Buffer Tank
Org N
NH4
NH4
NH3
CO2-containing gas (e.g. biogas or flue
NH3
BIOGAS (CH4 + CO2)

Fig. 20

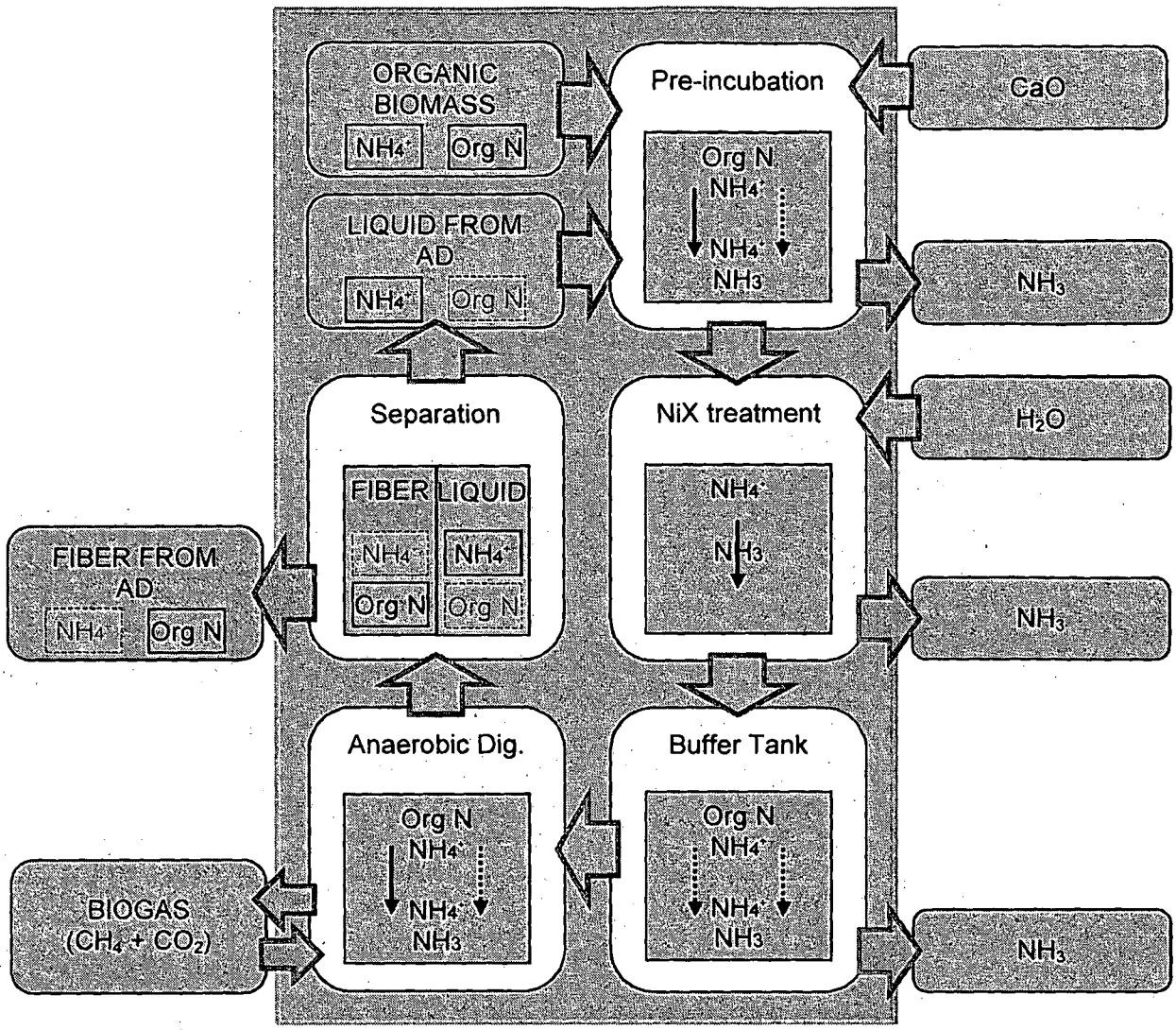

ORGANIC BIOMASS
NH4+
Org N
LIQUID FROM AD
NH4+
Org N
Pre-incubation
Org N
NH4+
NH4+
NH3
CaO
NH3
Separation
FIBER
LIQUID
NH4+
NH4+
Org N
Org N
NiX treatment
NH4+
NH3
H2O
NH3
FIBER FROM AD
NH4+
Org N
Anaerobic Dig.
Org N
NH4+
NH4+
NH3
Buffer Tank
Org N
NH4+
NH4+
NH3
BIOGAS
(CH4 + CO2)
NH3

Fig. 21

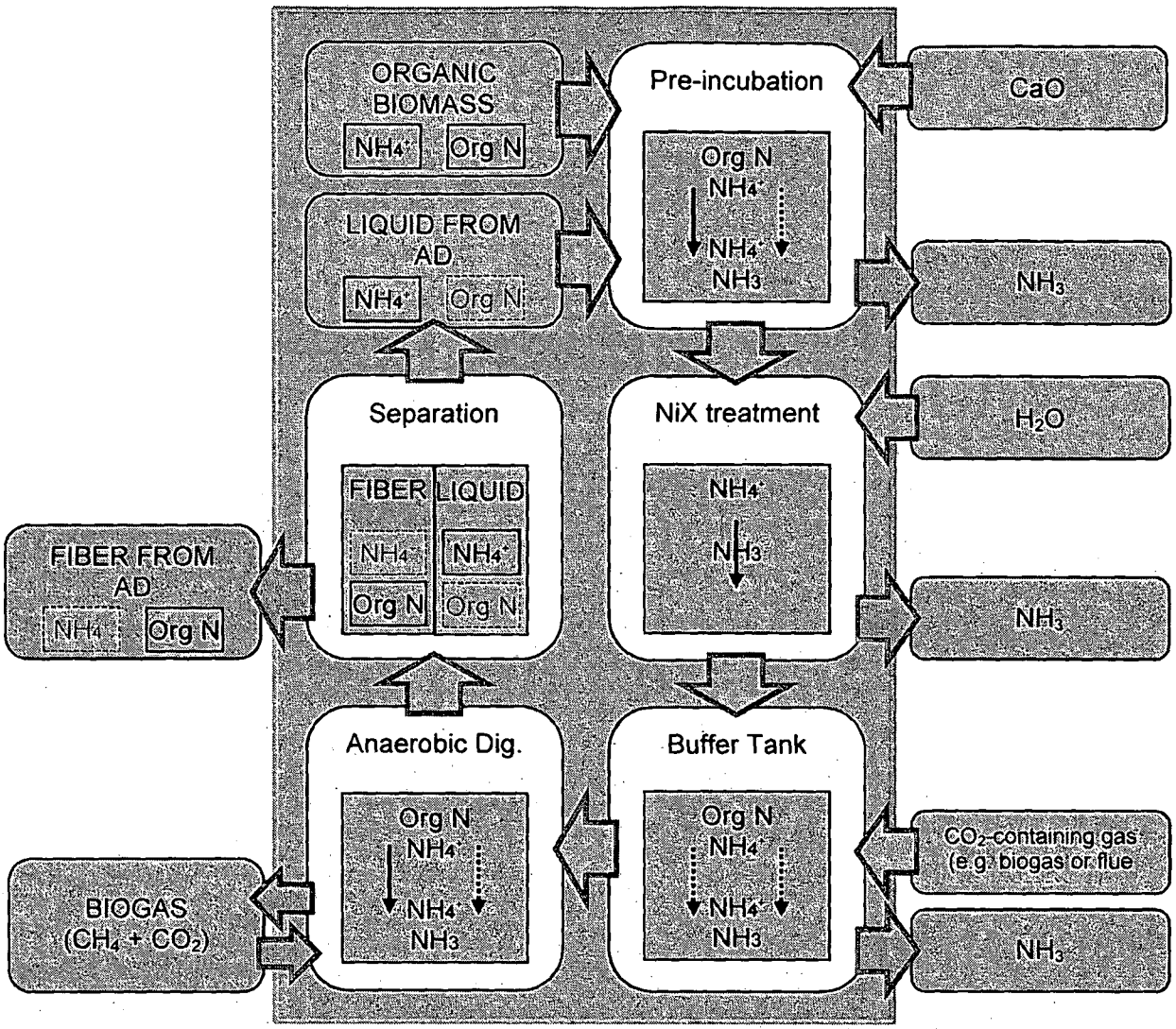

ORGANIC BIOMASS
NH4+ Org N
LIQUID FROM AD
NH4+ Org N
FIBER FROM AD
NH4+ Org N
BIOGAS (CH4 + CO2)
Pre-incubation
Org N
NH4+
NH4+
NH3
Separation
FIBER
LIQUID
NH4+
NH4+
Org N
Org N
NiX treatment
NH4+
NH3
Anaerobic Dig.
Org N
NH4+
NH4+
NH3
Buffer Tank
Org N
NH4+
NH4+
NH3
CaO
NH3
H2O
NH3
CO2-containing gas (e.g. biogas or flue
NH3

Fig. 22

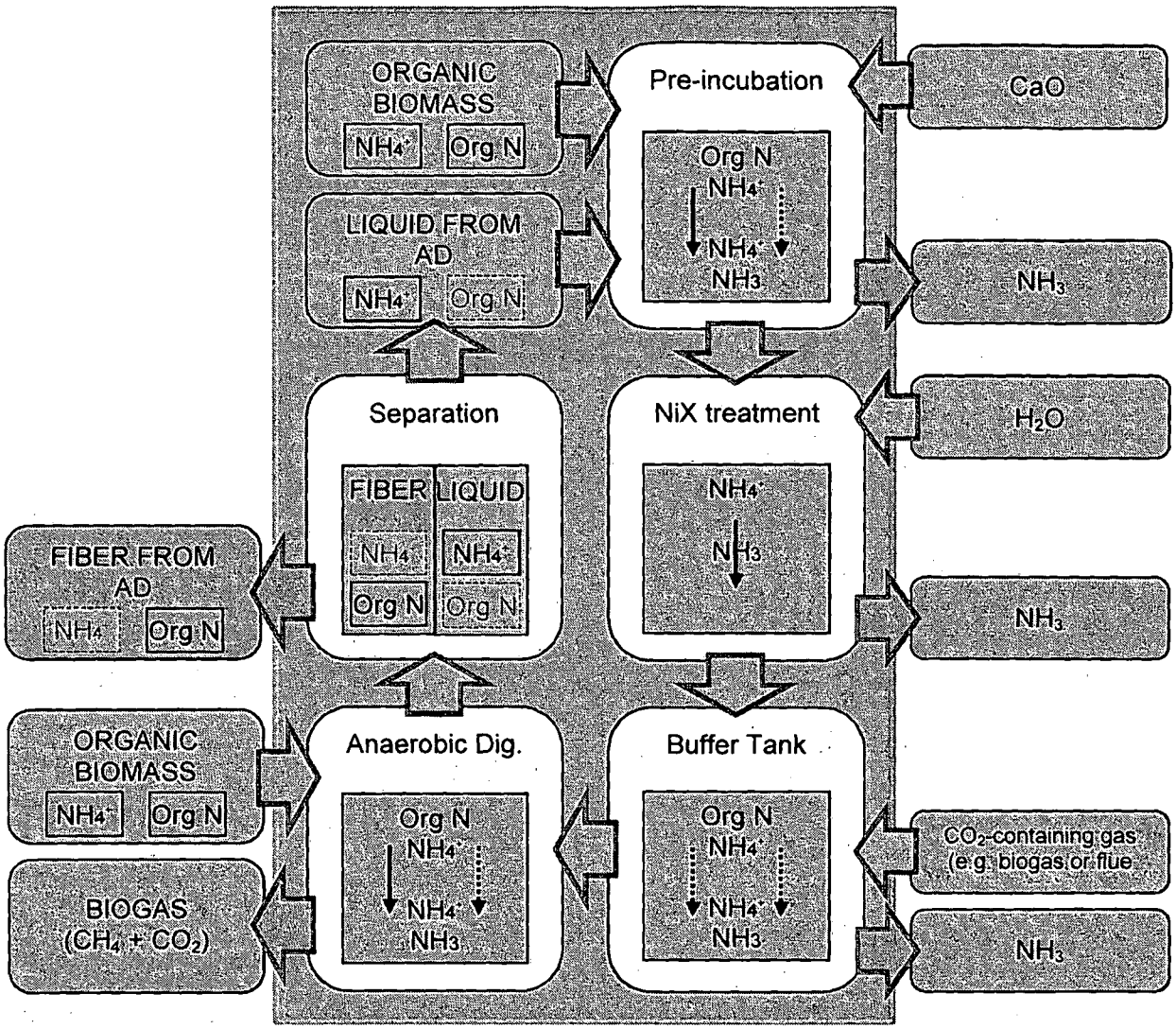

ORGANIC BIOMASS
NH4+ Org N
LIQUID FROM AD
NH4+ Org N
FIBER FROM AD
NH4+ Org N
ORGANIC BIOMASS
NH4+ Org N
BIOGAS (CH4 + CO2)
Pre-incubation
Org N
NH4+
NH4+
NH3
Separation
FIBER LIQUID
NH4+ NH4+
Org N Org N
NiX treatment
NH4+
NH3
Anaerobic Dig.
Org N
NH4+
NH4+
NH3
Buffer Tank
Org N
NH4+
NH4+
NH3
CaO
NH3
H2O
NH3
CO2-containing gas (e.g. biogas or flue
NH3

Fig. 23

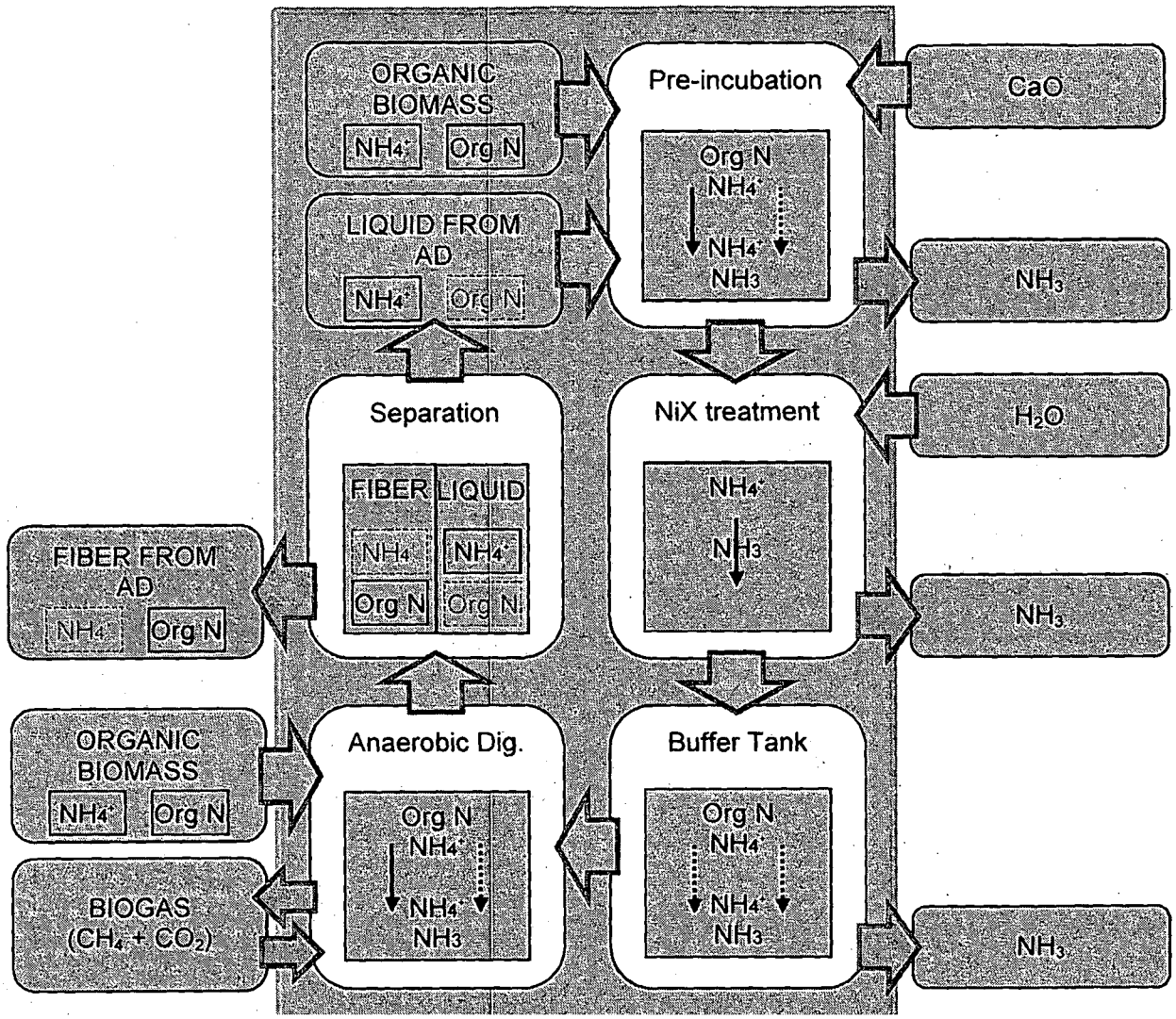

ORGANIC BIOMASS
NH4+ Org N
LIQUID FROM AD
NH4+ Org N
Pre-incubation
Org N
NH4+
NH4+
NH3
CaO
NH3
H2O
Separation
FIBER LIQUID
NH4+ NH4+
Org N Org N
NiX treatment
NH4+
NH3
FIBER FROM AD
NH4+ Org N
NH3
ORGANIC BIOMASS
NH4+ Org N
BIOGAS (CH4 + CO2)
Anaerobic Dig.
Org N
NH4+
NH4+
NH3
Buffer Tank
Org N
NH4+
NH4+
NH3
NH3

Fig. 24

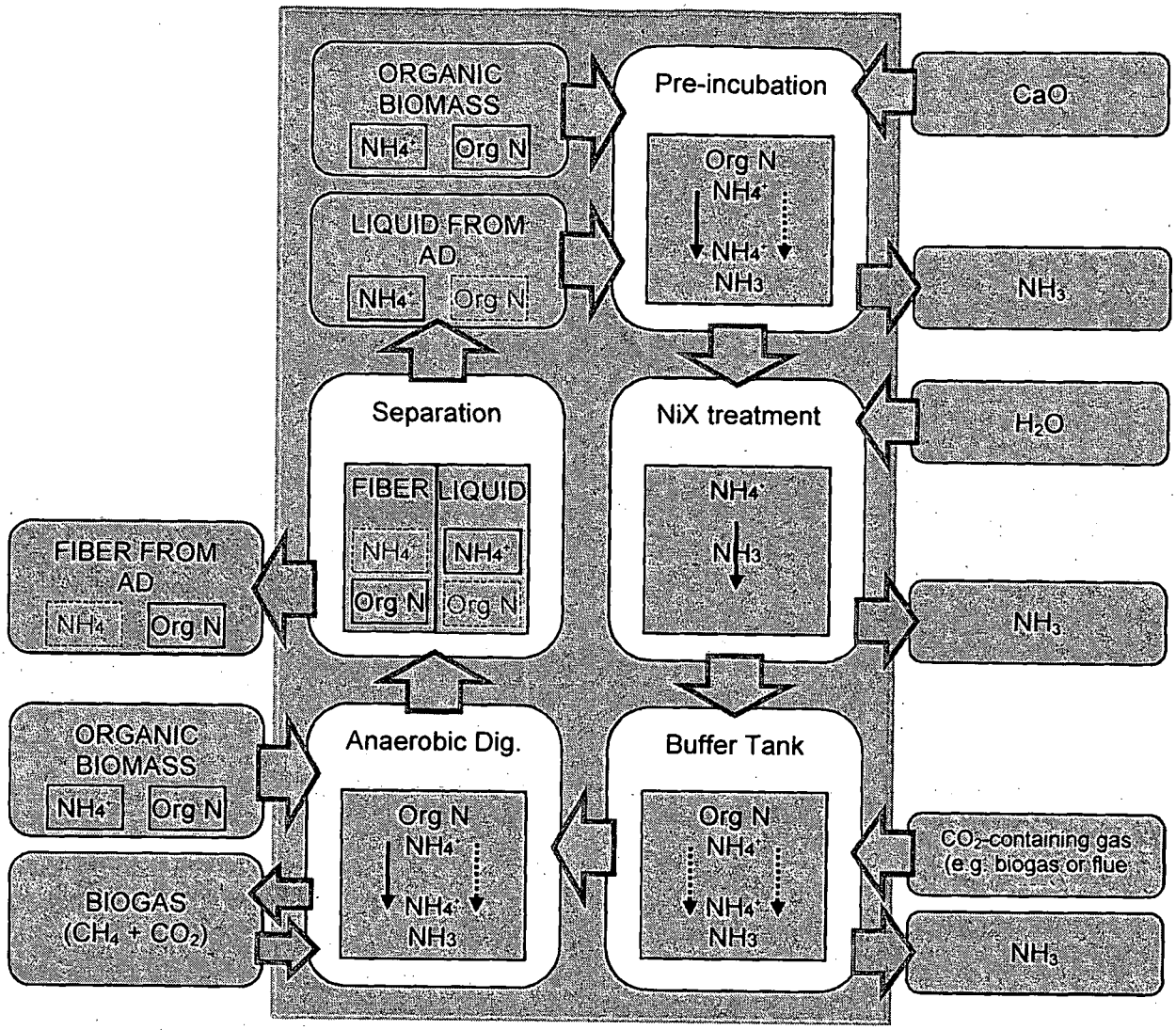
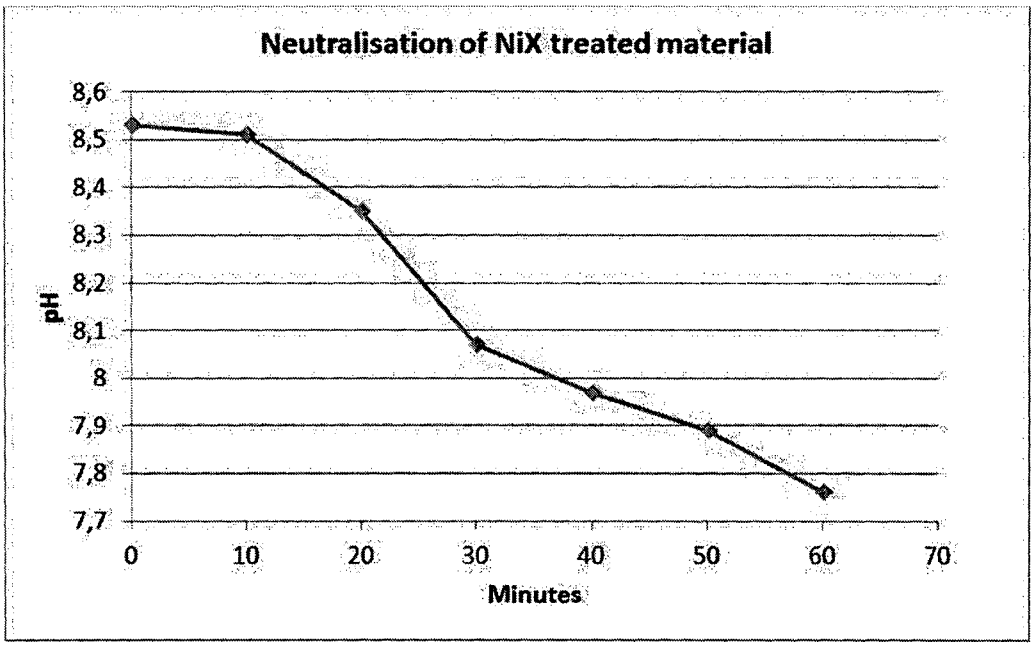

ORGANIC BIOMASS
NH4+ Org N
LIQUID FROM AD
NH4+ Org N
FIBER FROM AD
NH4+ Org N
ORGANIC BIOMASS
NH4+ Org N
BIOGAS (CH4 + CO2)
Pre-incubation
Org N
NH4+
NH4+
NH3
Separation
FIBER LIQUID
NH4+ NH4+
Org N Org N
NiX treatment
NH4+
NH3
Anaerobic Dig.
Org N
NH4+
NH4+
NH3
Buffer Tank
Org N
NH4+
NH4+
NH3
CaO
NH3
H2O
NH3
CO2-containing gas (e.g. biogas or flue
NH3

Fig. 25

Time response curve for pH neutralisation of lime pressure cooked (NiX treated) biomaterial



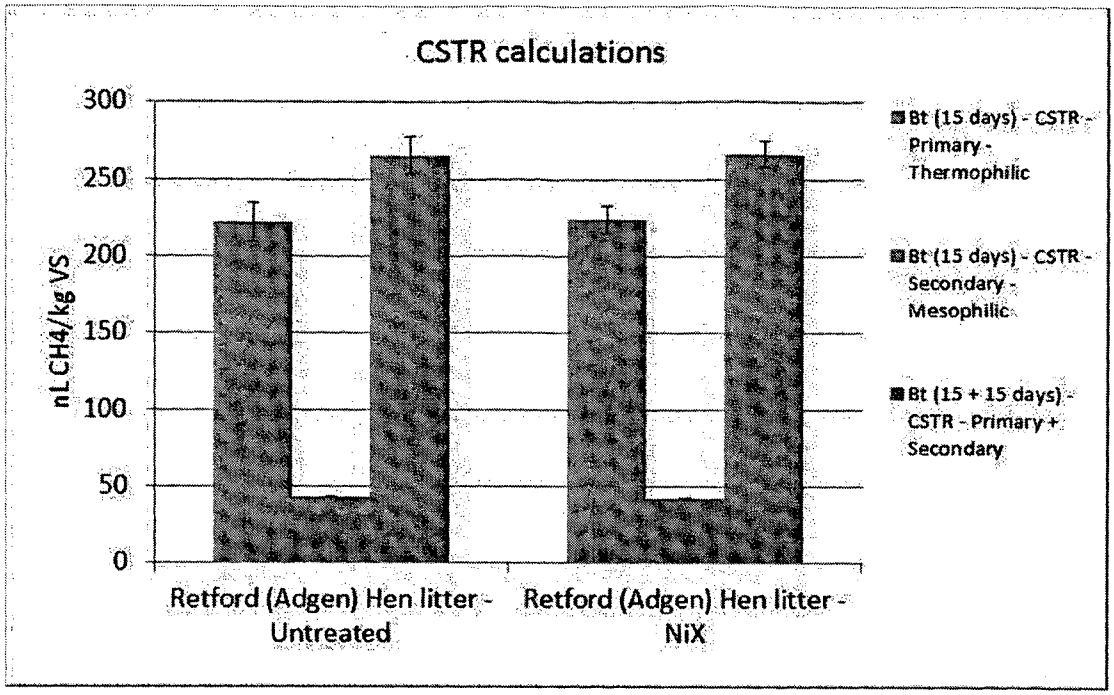


FIGURE 26

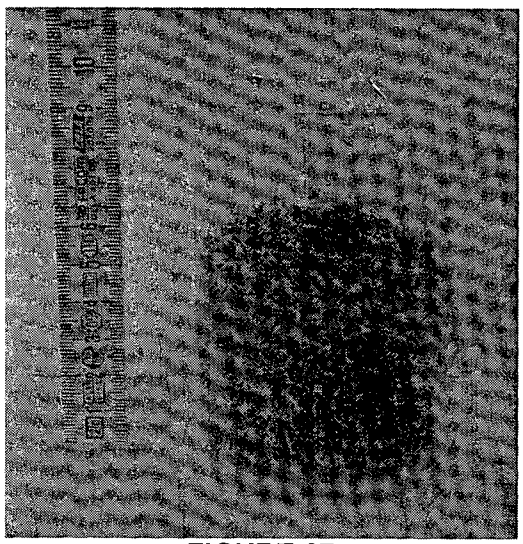


FIGURE 27

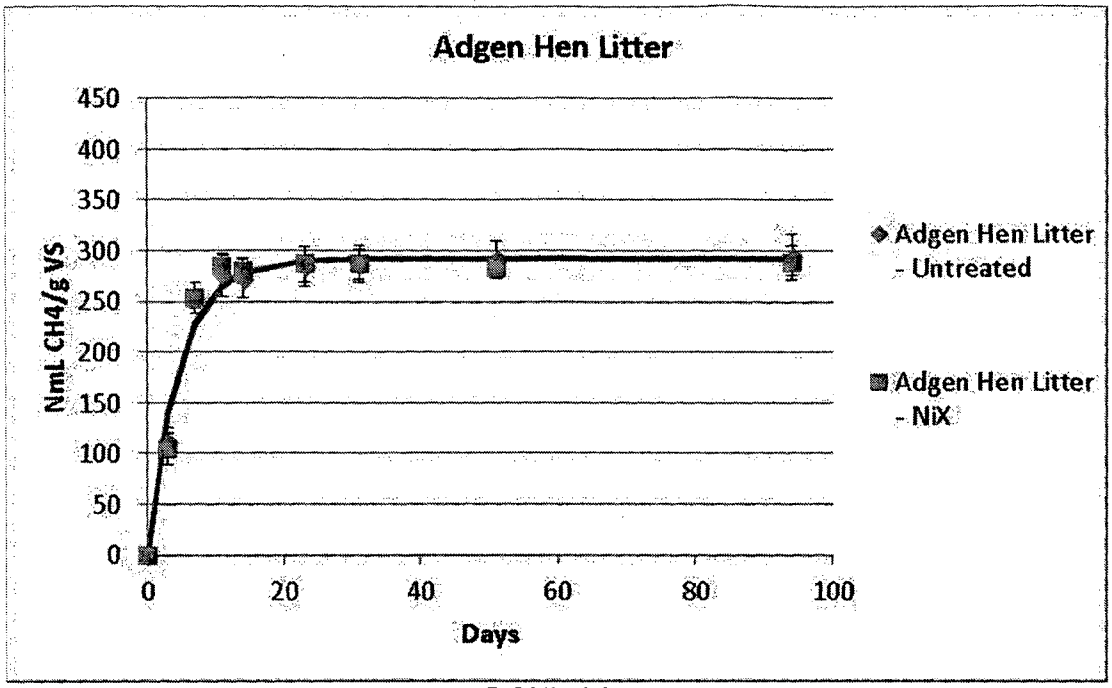


FIGURE 28

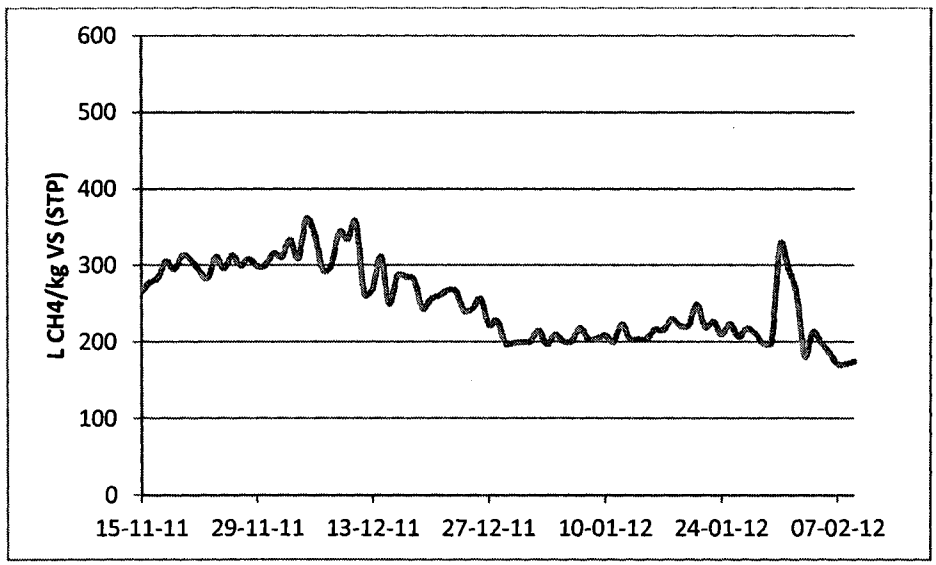


FIGURE 29

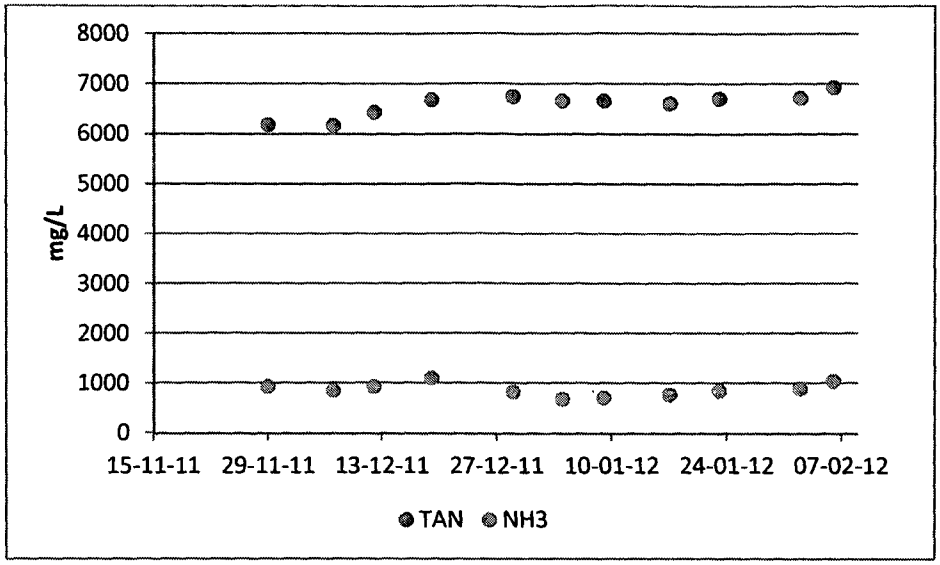


FIGURE 30

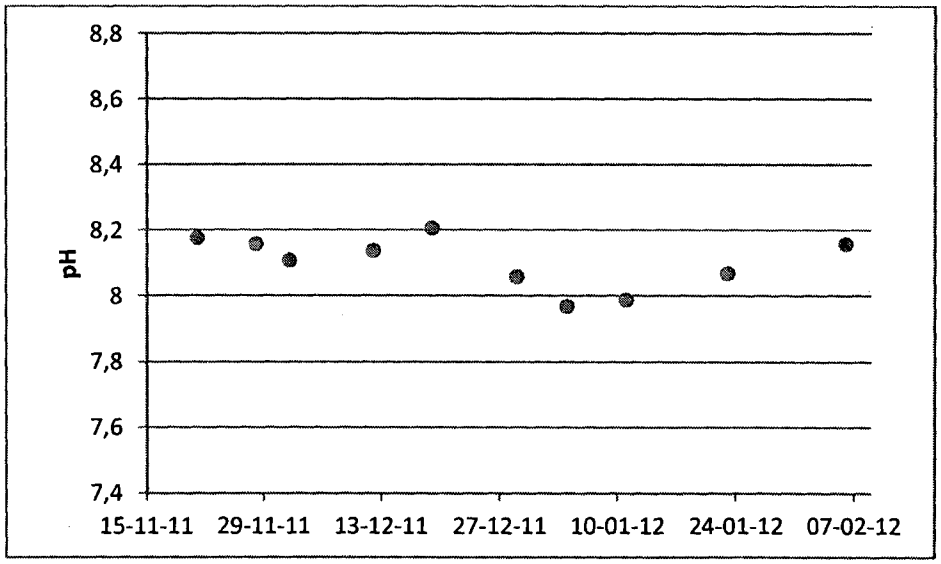


FIGURE 31

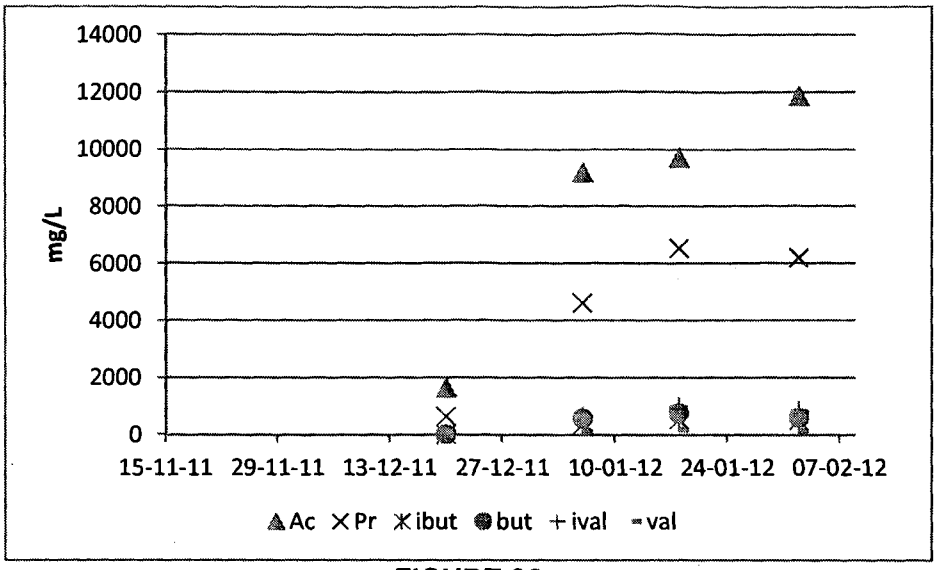


FIGURE 32

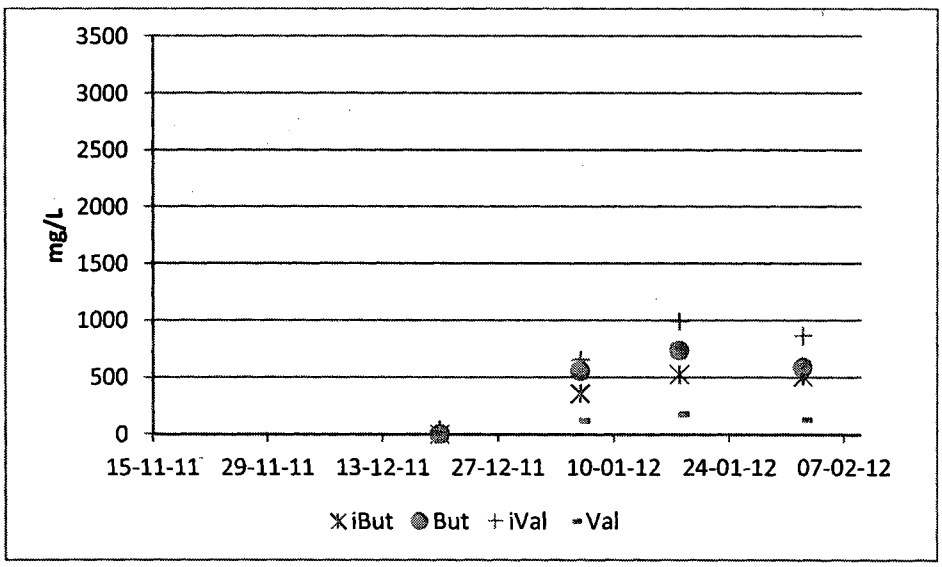


FIGURE 33

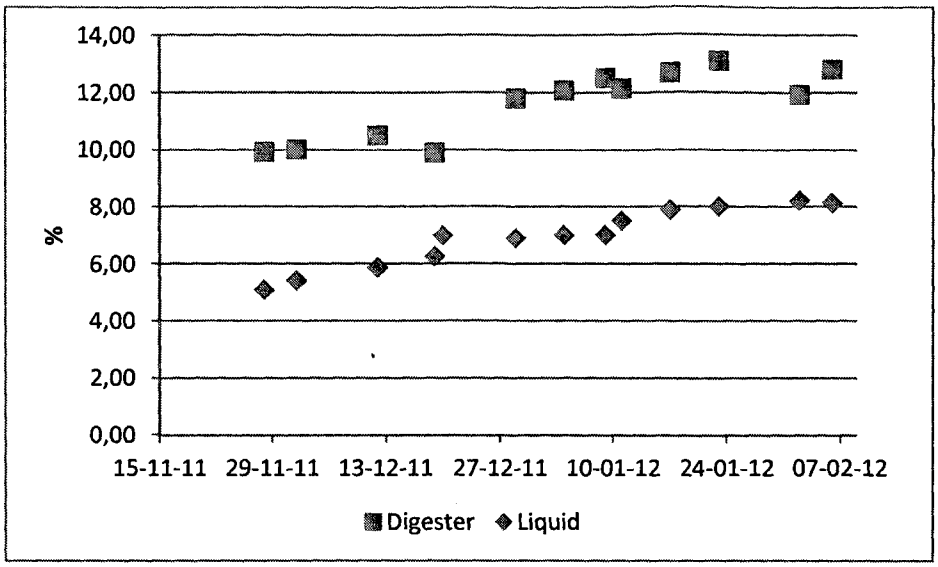


FIGURE 34

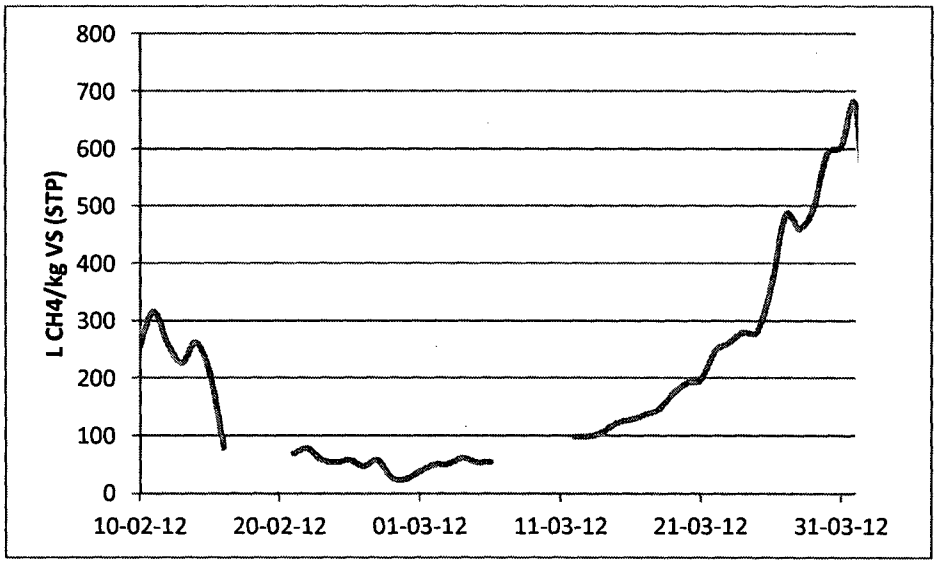


FIGURE 35

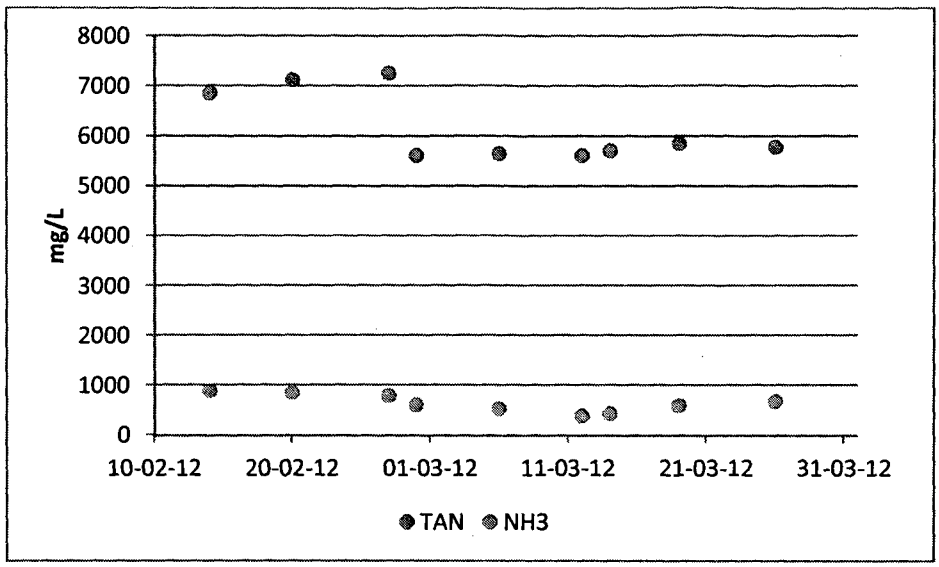


FIGURE 36

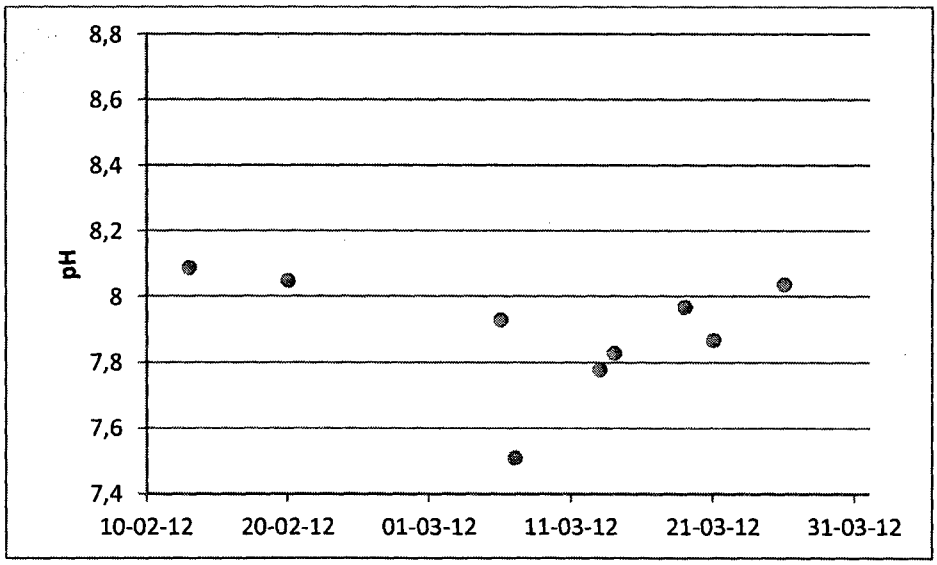


FIGURE 37

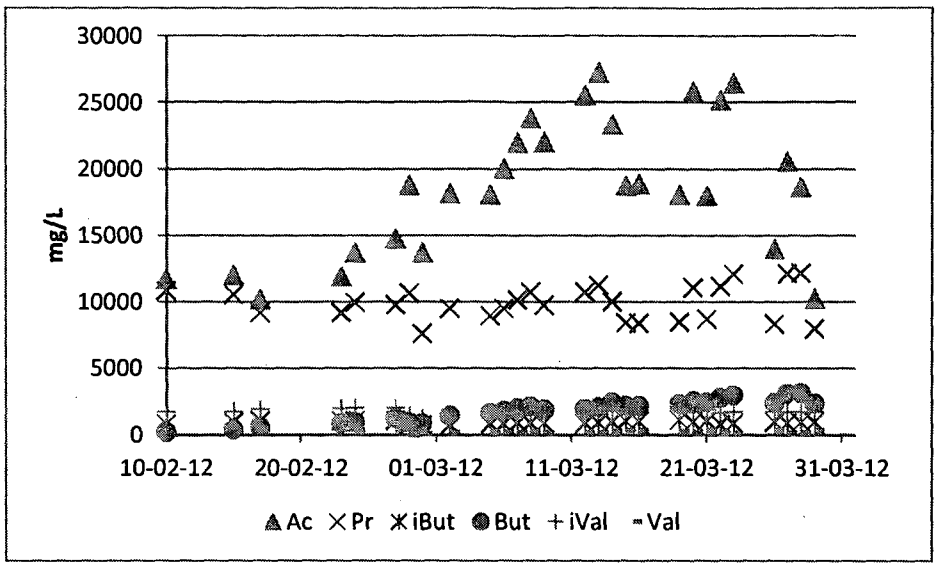


FIGURE 38

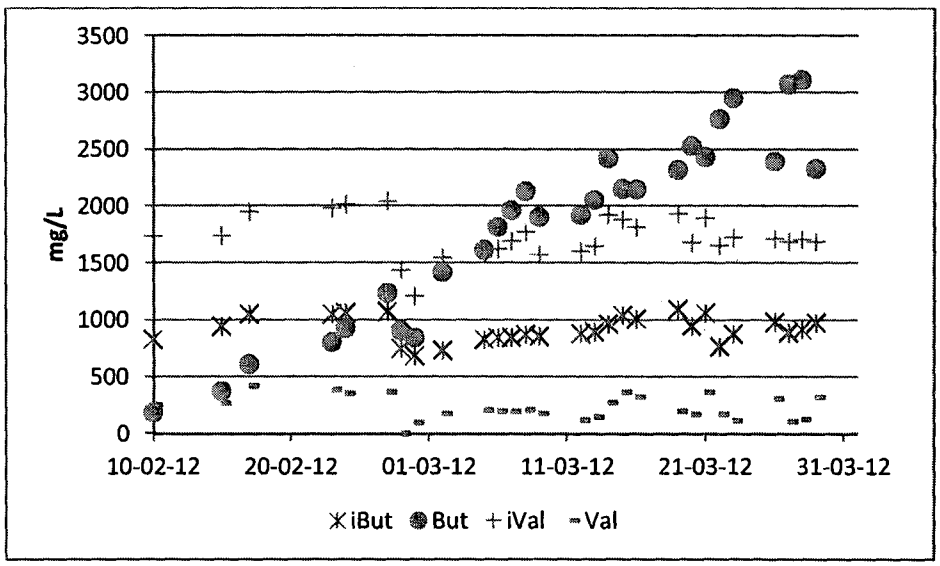


FIGURE 39

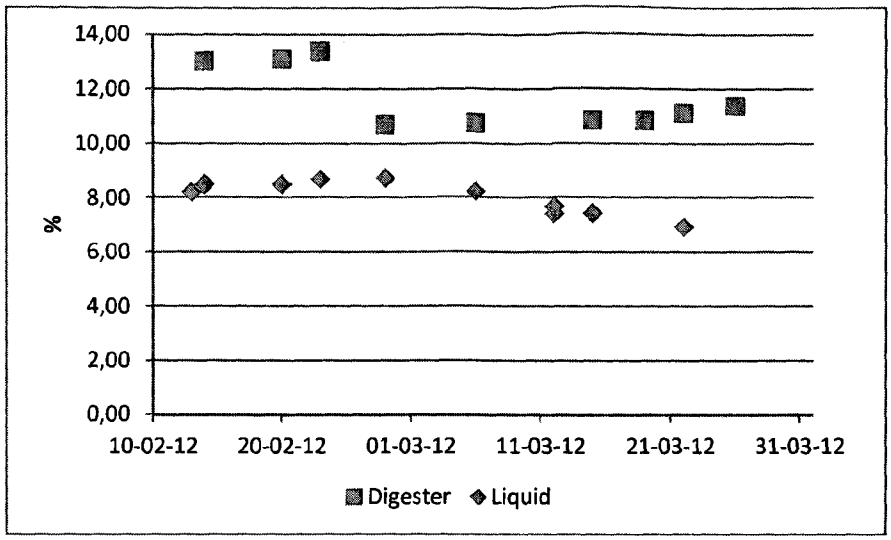


FIGURE 40

5

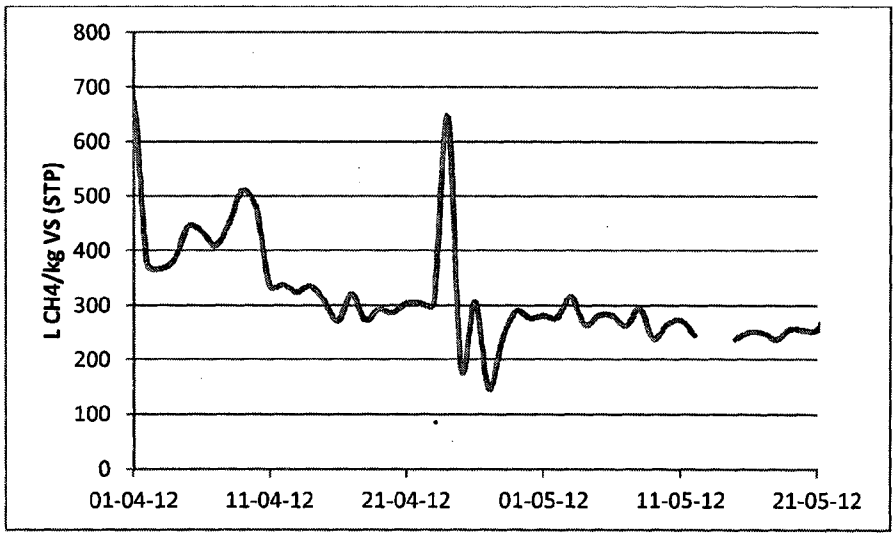


FIGURE 41

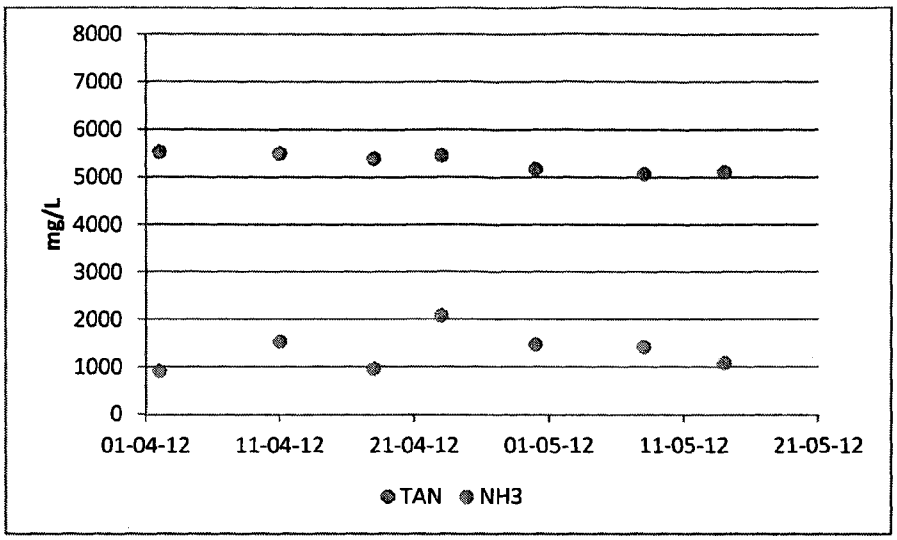


FIGURE 42

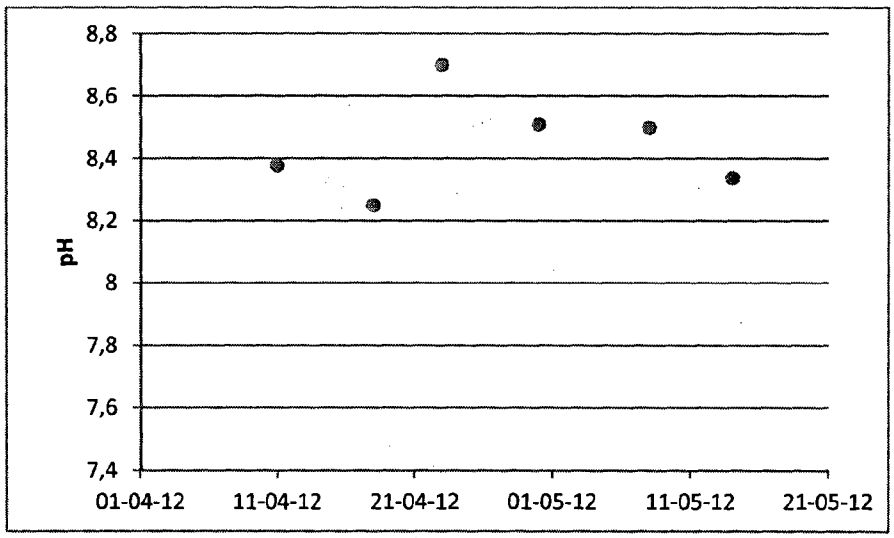


FIGURE 43

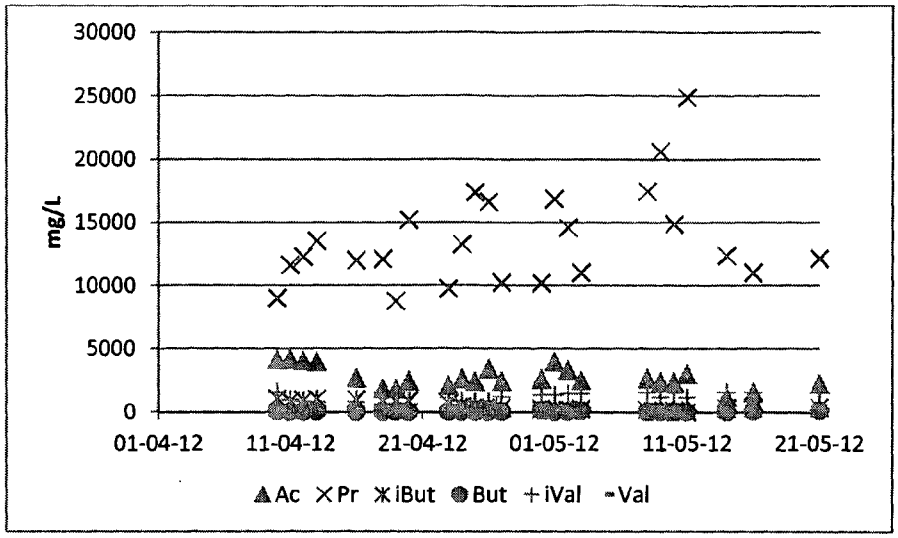


FIGURE 44

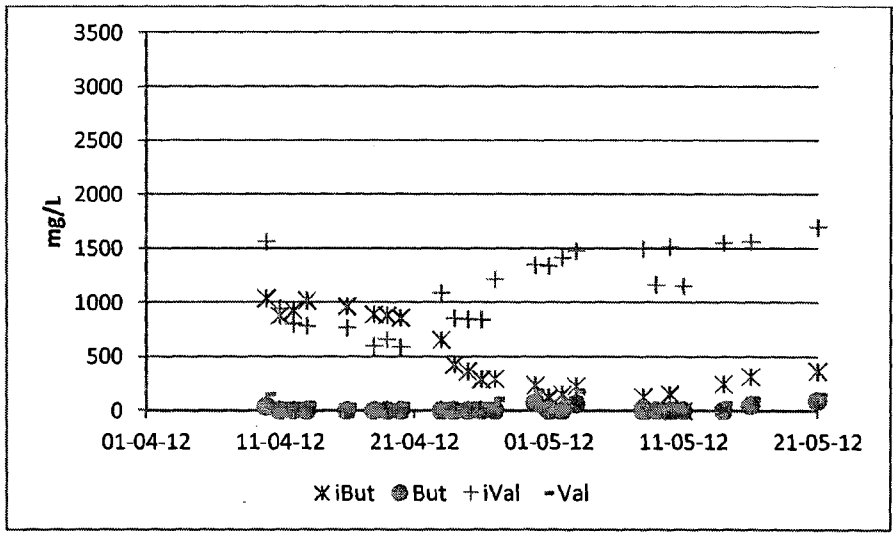


FIGURE 45

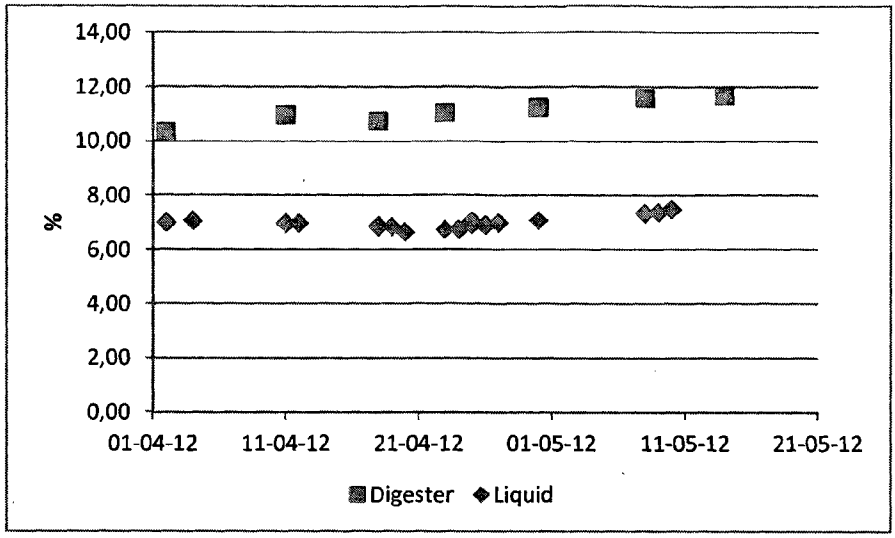


FIGURE 46

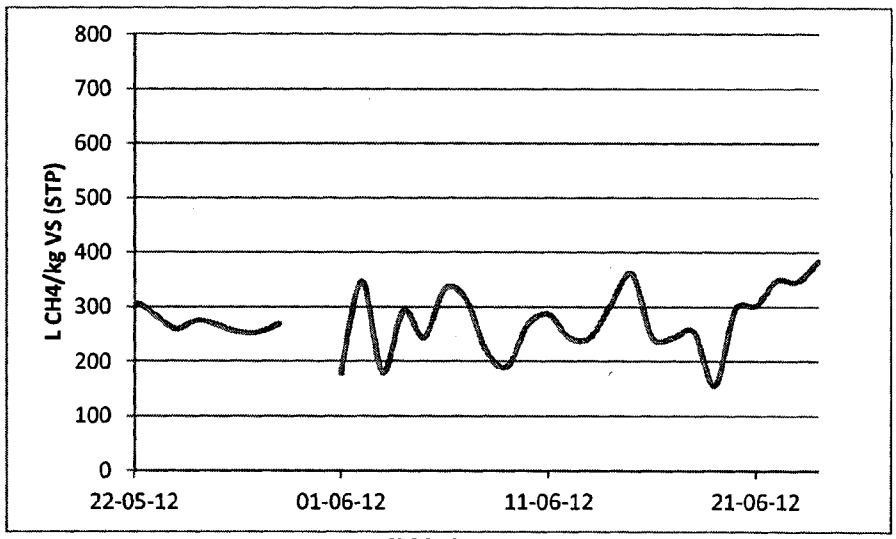


FIGURE 47

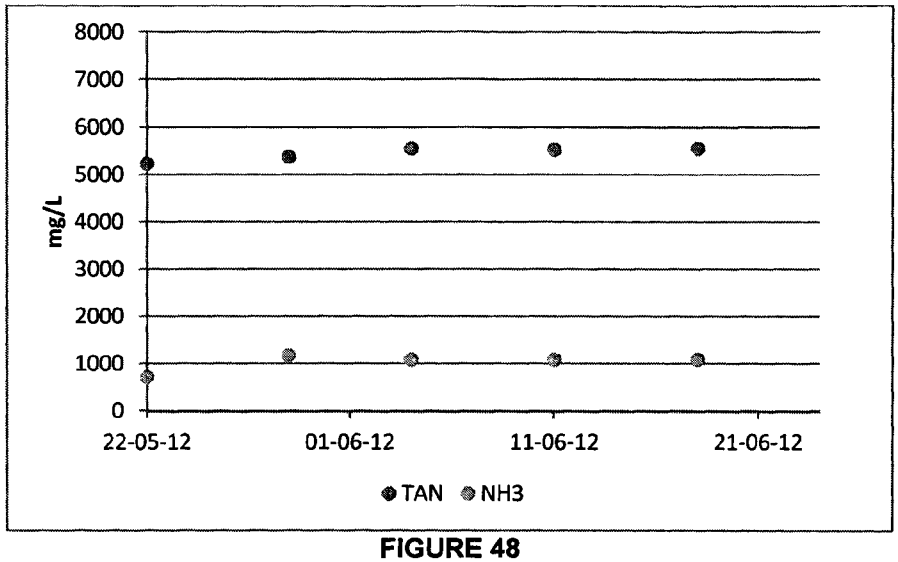


FIGURE 48

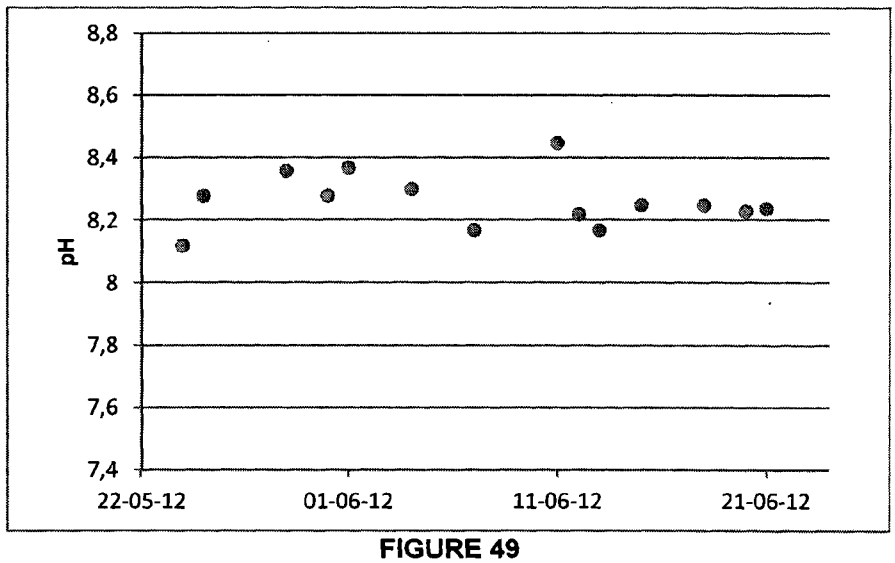


FIGURE 49

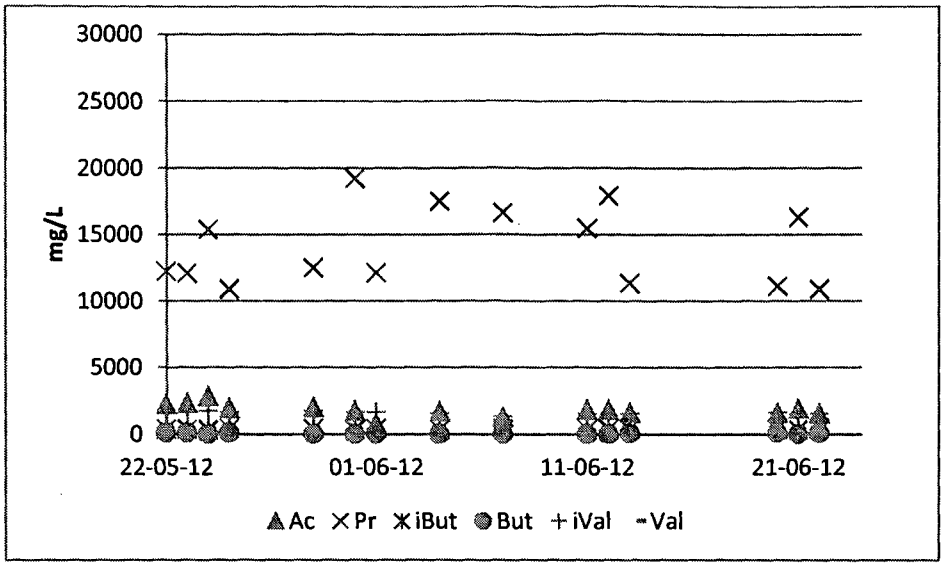


FIGURE 50

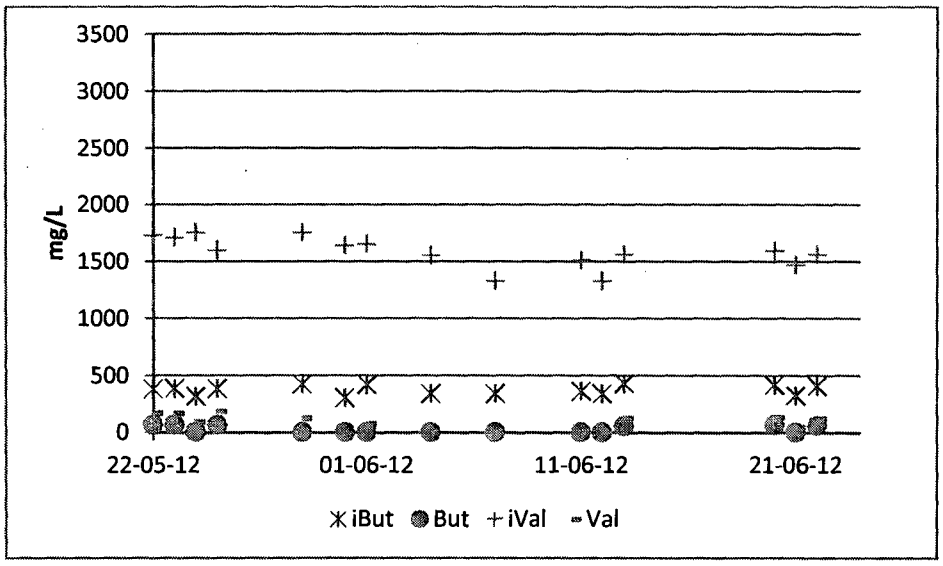


FIGURE 51

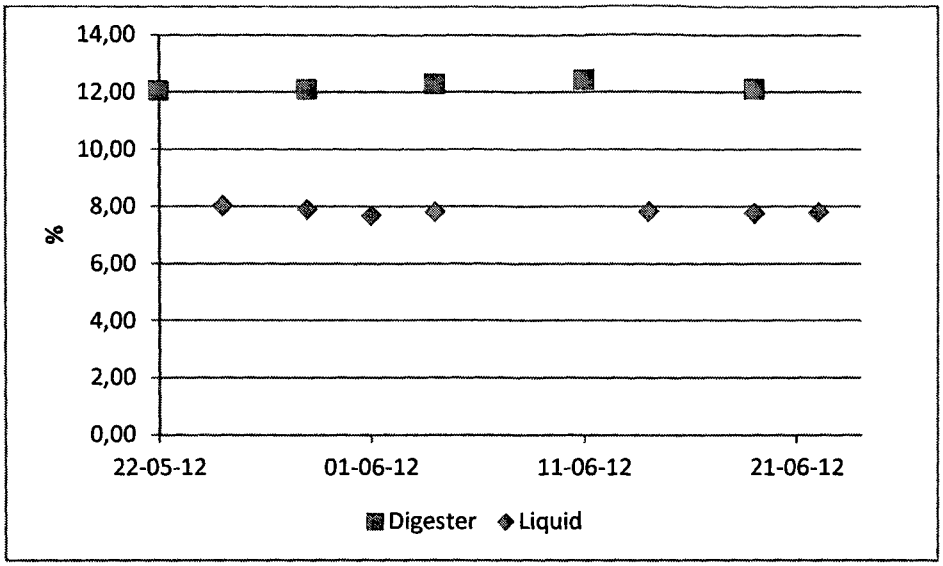


FIGURE 52

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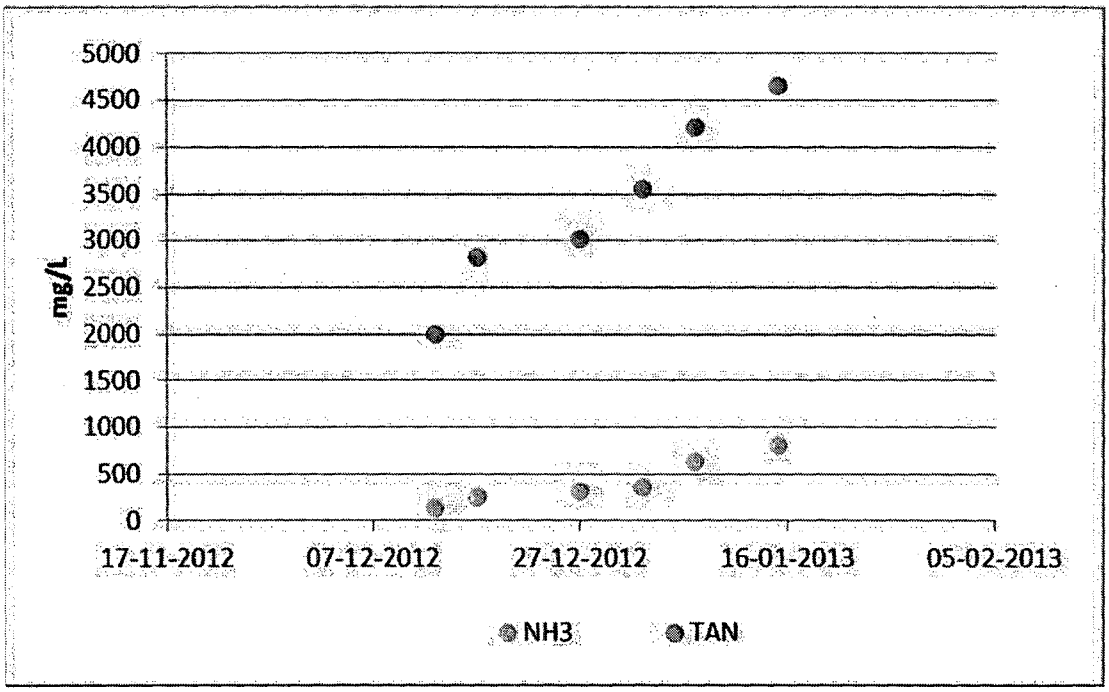


FIGURE 53

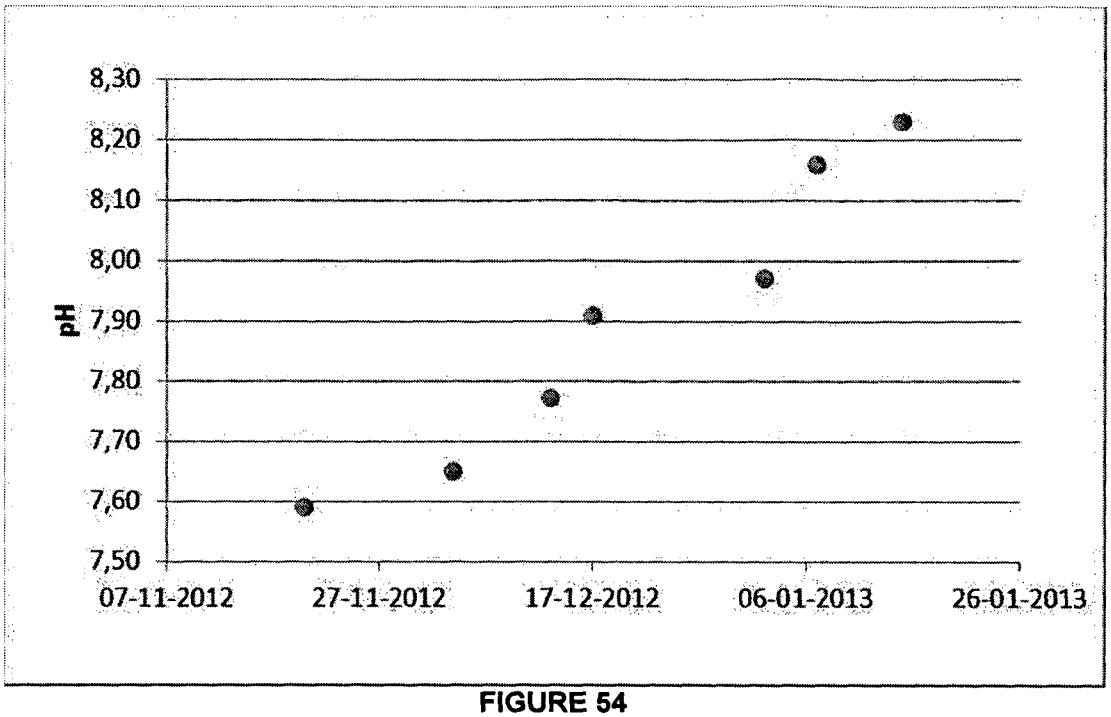


FIGURE 54

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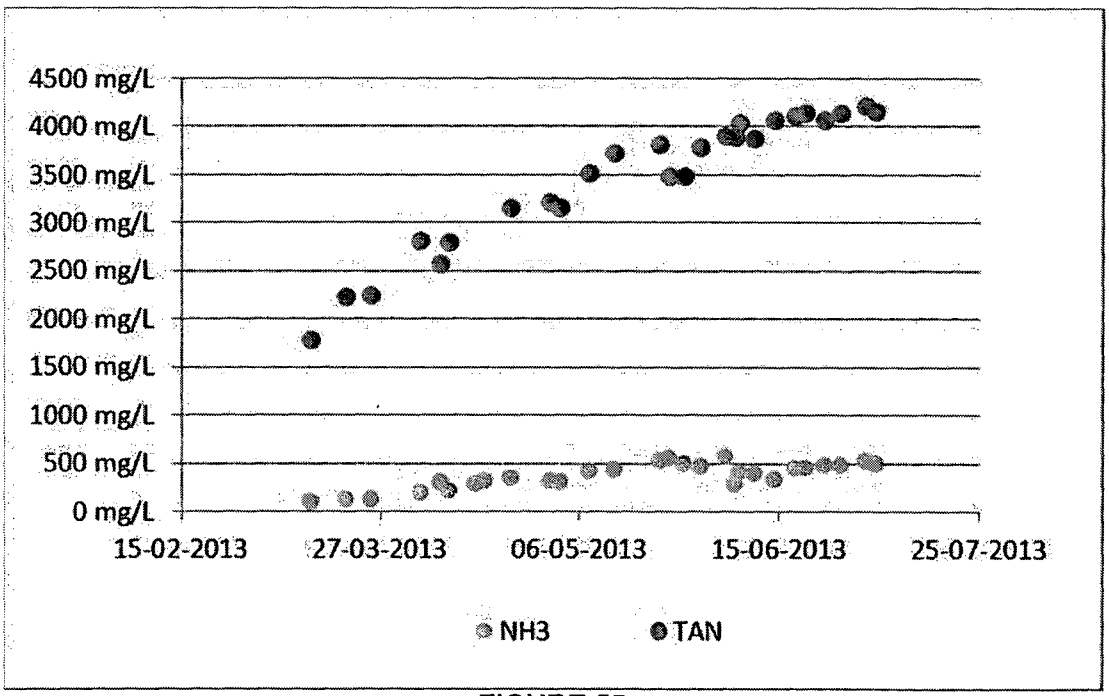


FIGURE 55

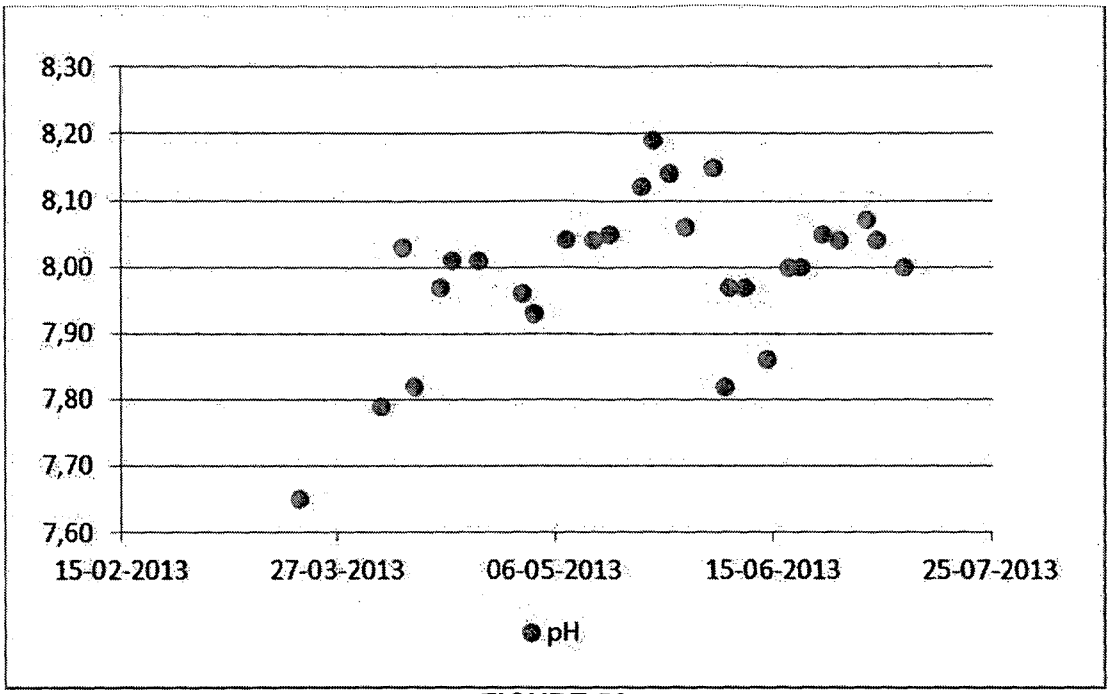


FIGURE 56

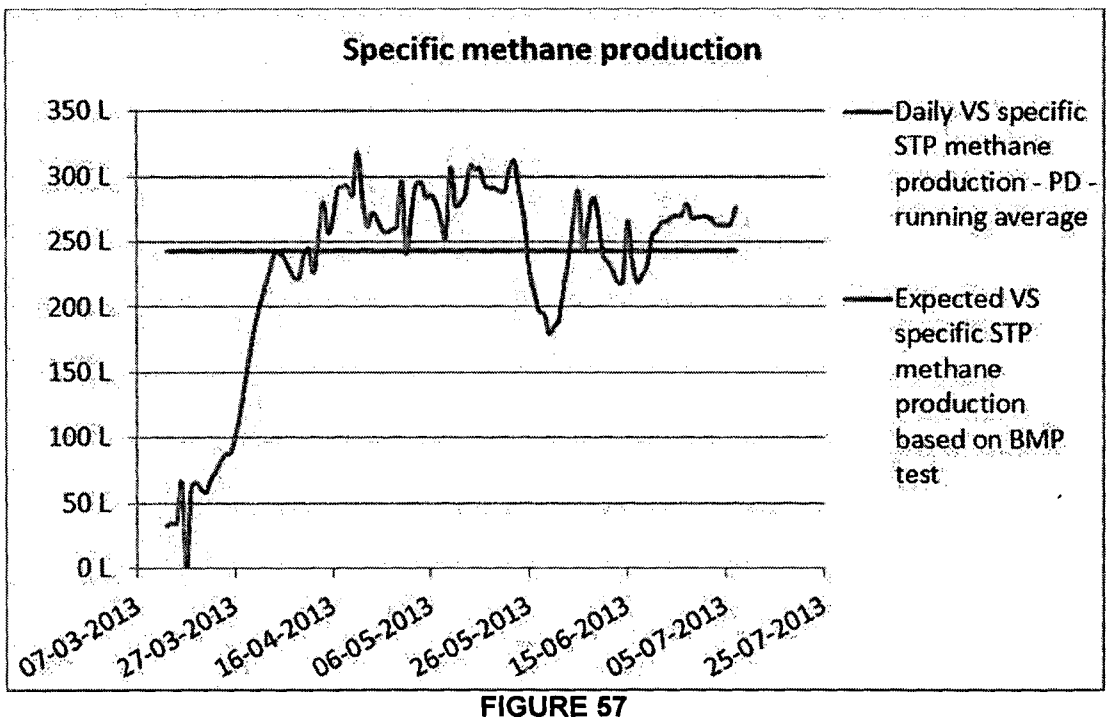


FIGURE 57

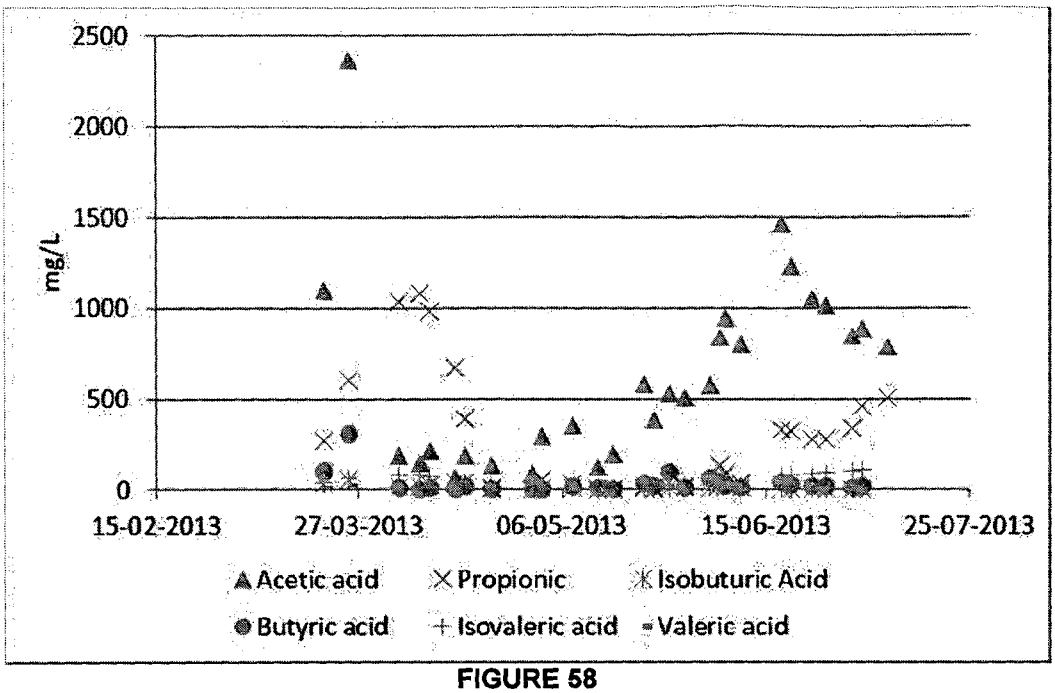


FIGURE 58

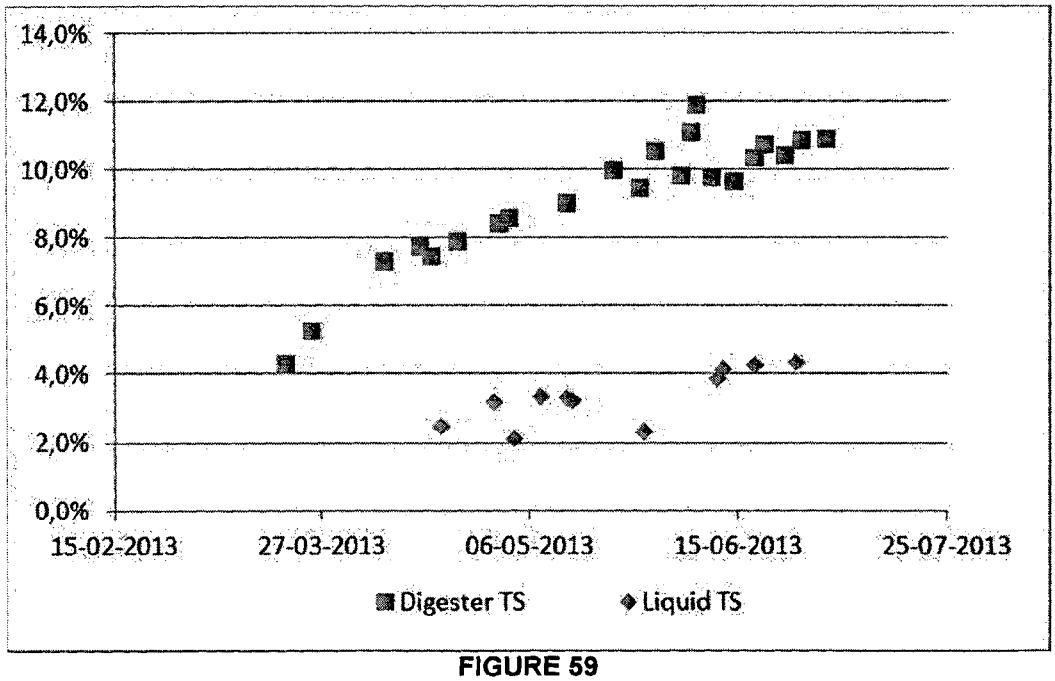


FIGURE 59

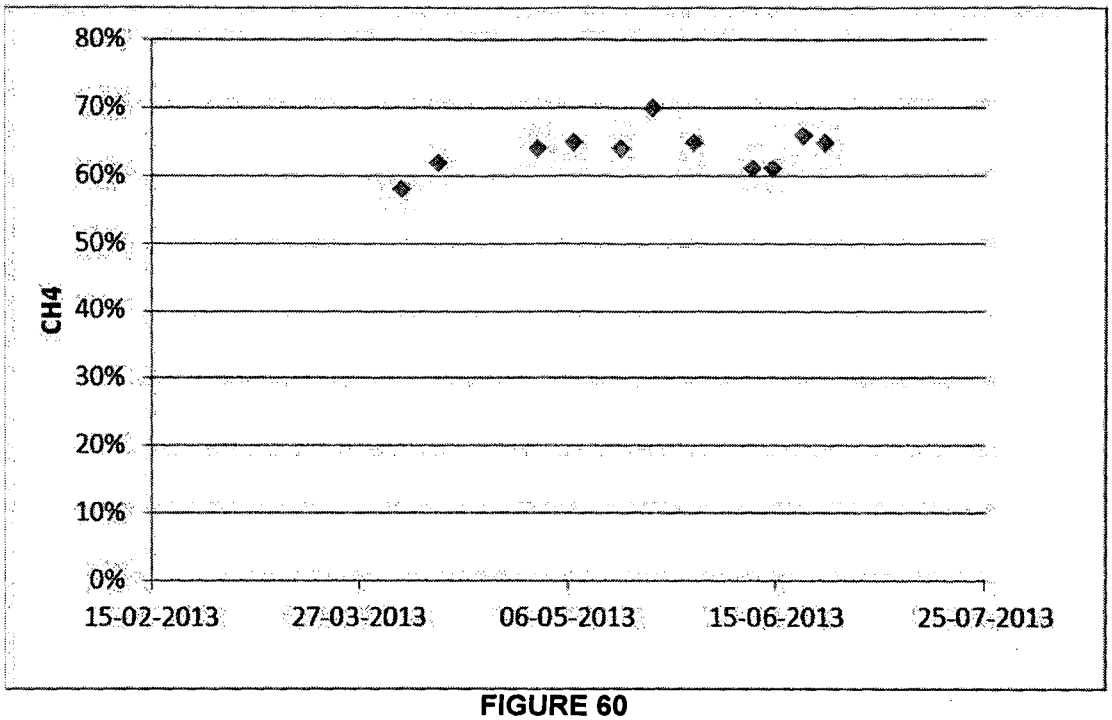


FIGURE 60

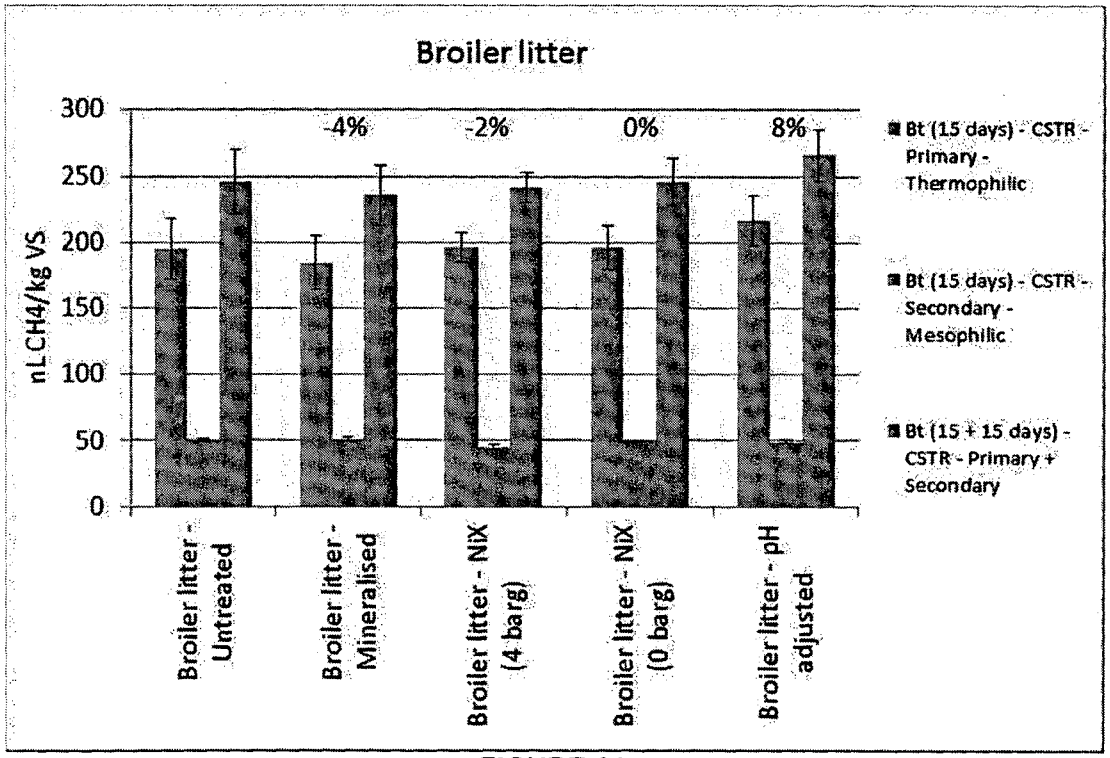


FIGURE 61

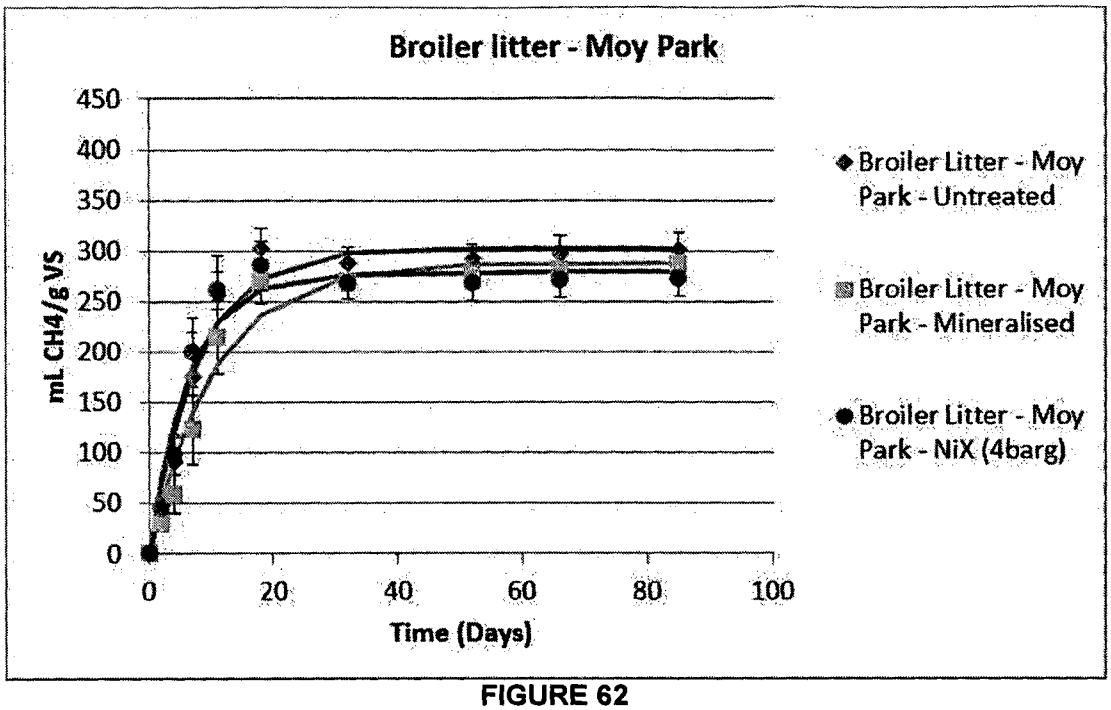


FIGURE 62

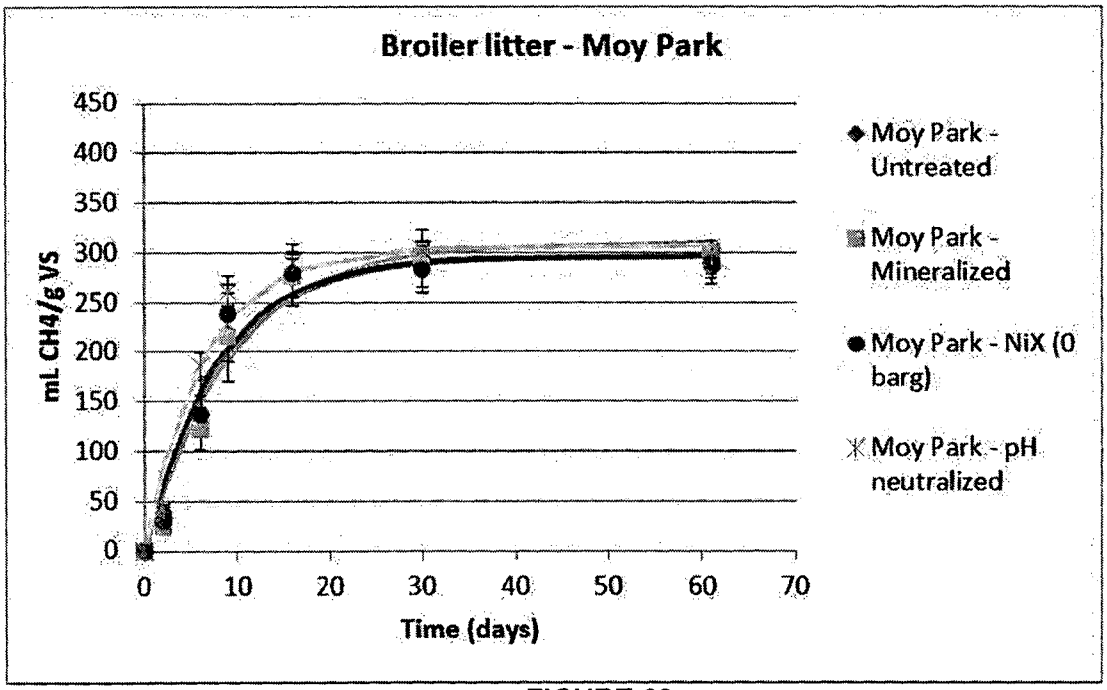


FIGURE 63



Fig. 64

Precipitated material during pH neutralisation using $CO_{2(g)}$

INTERNATIONAL SEARCH REPORT

International application No
PCT/DK2014/050116

A. CLASSIFICATION OF SUBJECT MATTER
INV. C12P5/00 C12P7/00 C02F11/04 C12M1/107
ADD.

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
C12P C02F C12M

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)
EPO-Internal, FSTA, BIOSIS, EMBASE

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 2004/025715 A1 (BONDE ET AL) 12 February 2004 (2004-02-12) * See the parts relating to lime pressure cooking, e.g. [0072, 0189, 0474, 0496, and 0555] * -----	1-78
A	JURADO ET AL: "Enhanced methane productivity from manure fibers by aqueous ammonia soaking pretreatment", APPLIED ENERGY, vol. 109, 25 April 2013 (2013-04-25), pages 104-111, XP028569744, * See page 104 (Abstract); early online publication * -----	1-78

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[X] Further documents are listed in the continuation of Box C.

[X] See patent family annex.

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"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

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Date of the actual completion of the international search	Date of mailing of the international search report
17 July 2014	31/07/2014

Name and mailing address of the ISA/	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Fax: (+31-70) 340-3016	Korsner, Sven-Erik

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C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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A,P	WO 2013/060338 A1 (XERGI NIX TECHNOLOGY A/S) 2 May 2013 (2013-05-02) * See pages 2-3 (Summary of the invention) and Figure 26 *	1-78
A,P	FINK: "How pre-treatment reduces contamination and aids digestion", The Anaerobic Digestion & Biogas Association's Annual Expo & Conference; Birmingham, 3-4 July 2013, July 2013 (2013-07), pages 1-17, XP002727307, Retrieved from the Internet: URL:http://www.adbiogas.co.uk/wp-content/uploads/2013/07/Jorgen-Fink.pdf [retrieved on 2014-07-16] * See pages 3, 4, 5, 6, 7, 8, 9, 10, 11 *	1-78
A,P	JIANG ET AL: "Evaluation of an integrated ammonia stripping, recovery, and biogas scrubbing system for use with anaerobically digested dairy manure", BIOSYSTEMS ENGINEERING, vol. 119, 25 February 2014 (2014-02-25), pages 117-126, XP028625487, * See sections 2.6 and 3.4-3.5; early online publication *	1-78

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Information on patent family members

International application No
PCT/DK2014/050116

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