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ORIGINAL RESEARCH

Stripping and scrubbing of ammonium using common fractionating columns to prove ammonium inhibition during anaerobic digestion

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Abstract
Anaerobic digestion to produce biogas is generally considered as one of the most sustainable technologies for the production of renewable energy. During this microbial process, organically bound nitrogen is released as ammonium that ends up in the digestate and finally may inhibit the process. In this study, it is investigated if ammonium can be removed and recovered out of the liquid fraction of a thermophilic digestate from a potato processor. This is achieved at laboratory scale through an easy and self-designed stripping and scrubbing process using Vigreux and Dufton columns, which are commonly used laboratory fractionating columns. The stripping is performed at pH 8.5 and at 323.15 K (50 °C), which results in the volatilization of the ammonium present in ammonia. Subsequently, the stripping gas charged with ammonia is put into contact with a sulphuric acid solution, resulting in $(NH_4)_2SO_4$, which can be used as an N–S fertilizer. In addition, the digestion experiments have demonstrated that the biogas yield is 36% higher after removal of the ammonium from the digestate compared to the untreated digestate.

Keywords Ammonium · Anaerobic digestion · Biogas · Stripping and scrubbing

Introduction

The anaerobic digestion of organic waste is generally considered as one of the most sustainable technologies to produce renewable energy. The biogas produced can replace conventional fuels to generate heat and power [1–5]. Anaerobic digestion is a natural microbial process in which microorganisms break down complex organic matter in the absence of oxygen. Although it is generally considered a two-phase process, it can be subdivided into various metabolic steps with the participation of several microbial groups, namely hydrolysis, acidogenesis, acidification and finally methanogenesis in which methane is formed [6]. The final product, biogas, is a mixture of methane (50–75%), carbon dioxide (25–50%) and other trace gases such as nitrogen (0–10%), hydrogen (0–1%), hydrogen sulphide (0–3%) and traces of oxygen [7]. Due to the presence of rigid and not readily degradable material, the digestion process proceeds sometimes slowly, which requires a long residence time (even up to 30 days) in the digester [8]. Even with these residence times, the conversion efficiency varies around 70–90%. In some cases, however, only approximately 50% of the total organic dry matter is converted into biogas, indicating that the digestate still contains a substantial amount of organic matter [8, 9].

The biogas sector in Flanders (Belgium) is currently under pressure due to the reduction of governmental grants and the recent decrease in electricity prices. Intervention in the digestion process leading to a higher biogas production efficiency is one of the primary measures to reduce costs, to further expand the sector and thus to stimulate green energy production by means of biogas. There are two main technical reasons for the limited conversion efficiency that needs to be addressed: (1) the limited conversion of organic material into biogas due to the presence of hardly biodegradable

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material and (2) the inhibition of the digestion process due to the presence of an increased concentration of ammoniacal nitrogen in the digester, as organically bound nitrogen is released as ammonium during anaerobic digestion. High concentrations (1.7–6.7 g L^{-1}) of total ammoniacal nitrogen (TAN) inhibit or are even toxic for methanogen bacteria which can cause a decrease in biogas production [10, 11].

From this point of view, it would be very interesting to reduce the TAN content of the digestate. This TAN reduction can be achieved through biological, chemical or physical processes [12]. Biological processes, in which classical nitrification–denitrification is most often applied, are in fact removal techniques since they convert the ammonium present into nitrogen gas. Furthermore, the conditions for nitrification will also result in oxidation of the available organic carbon and thus will result in loss of biogas potential. Chemical and chemical-physical processes for ammonia removal can be rather classified as recovery techniques because they give rise to the precipitation of, e.g. struvite ($MgNH_4PO_4{\cdot}6H_2O$) from raw digestate or its supernatant [13]. However, such recovery techniques have their drawbacks, such as high cost of chemicals and the need to strictly control the pH [14]. Another technique for ammonium recovery can be performed via a stripping and scrubbing process. Since the digestate of an anaerobic digestion has both a high temperature and a high TAN content, it is very suitable for ammonia stripping [15]. Ammonia stripping can be realized by using air, steam or biogas to separate the gaseous NH_3 from the liquid phase. The whole process depends on pH, temperature and mass transfer area. Next, the stripping gas, which is saturated with NH_3, is brought into contact with an acid solution (usually sulphuric acid). When sulphuric acid is used to capture ammonia, an $(NH_4)_2SO_4$ solution is formed [16]. Optionally, the more expensive nitric acid can also be used to capture ammonia that will produce NH_4NO_3, which is however more interesting for fertilization.

The key to an efficient stripping process lies in the design of the contact system between the TAN-rich digestate and the gas to strip out the NH_3. The aim is to maximize the level of contact while minimizing the energy costs. The most common stripping systems use continuously packed towers [17]. Other options are spray towers, low profile units, bubble diffusers, aspirators, surface aerators and high-intensity mixers. Selection of the appropriate technology is both case and site specific. Limoli et al. [18] investigated ammonia removal from raw manure digestate by means of a turbulent mixing stripping process. Batch tests demonstrated that sodium hydroxide was the most suitable alkaline chemical to control pH, as it is easy to handle and minimizes treatment time and costs. When NaOH was applied to treat raw digestate at pH 10 and a temperature of 296.15 K (23 °C), TAN removal efficiency reached 89% after 24 h of turbulent mixing stripping. Provolo et al. [19] investigated the performance of a stripping process based on a new concept of installation in which slow-rate NH_3 volatilization was promoted in a closed reactor containing continuously mixed digestate. Jiang et al. [20] investigated ammonia removal through air stripping of digested dairy manure using packed columns. Temperature and pH were identified as sensitive parameters for the process. When the temperature of the digestate was maintained at mesophilic level (308.15 K or 35 °C) during the stripping process, the optimized pH for ammonia stripping was 10.3 resulting in 90% ammonia removal. In various studies, self-built laboratory setups were used to strip ammonium from wastewater or digestate. Yuan et al. [21] demonstrated the removal of ammonia from wastewater using a continuous-flow rotating packed bed as an air stripper at ambient temperature. Bousek et al. [22] studied the impact of strip gas composition on side stream ammonia stripping of digestate. For this purpose, they used a laboratory-scale batch stripping and scrubbing plant constructed from glass elements. The stripping unit was equipped with a fast-rotating pedal at the upper end that functioned as a foam destruction device. The stripper was connected to the scrubber unit via a horizontal laboratory glass tube. The scrubber column (Vigreux column) and a 1 L round bottom flask were operated in parallel current mode using 1 M H_2SO_4 as scrubbing media.

In our present study, which is actually a continuation of our previous research [23], it was examined if such a stripping and scrubbing process can be applied to selectively remove and recover the TAN present in the thermophilic digestate from a potato processor. For this purpose, a self-developed laboratory setup is built, which initially used Vigreux columns, a commonly used laboratory fractionating column. To create an intense contact between the gas and liquid phases during stripping and scrubbing, the digestate and the acid solution, respectively, were recirculated over Vigreux columns, while an airstream was used to transport the ammonia from the stripping to the scrubbing part of the installation. Comparing treated (ammonium removed via stripping) and untreated digestate (no ammonium removal) allowed us to verify if the ammonium present in this particular digestate really exerts an inhibitory effect on the biogas production or not. In a next phase, the stripping and scrubbing process was further investigated by testing different combinations of a Vigreux column and a Dufton column, the latter being another type of fractionating column.

Experimental

To investigate the possible inhibitory effect of TAN during anaerobic digestion, it is necessary to selectively remove this component from the digestate without deactivating the microorganisms present. Such ammonium removal can be effectively performed with a stripping and scrubbing process

at moderate conditions. From a stripping prospective, it is designated to separate the raw digestate into a solid (thick) and a liquid (thin) fraction. The latter has better characteristics than the raw digestate due to the lower solid content. Studies pointed out that a higher solid content exhibits a lower nitrogen removal efficiency than those with low solid content [15, 24]. The experimental procedure used in this study is schematically represented in a flowchart (Fig. 1).

The digestate used in this study is taken from a thermophilic digester that is fed with potato waste. Its origin and characterization are explained in Sects. "Origin of the digestate and substrate" and "Characterization of the thermophilic digestate and substrate", respectively. As mentioned, the raw digestate is separated into a thick fraction and a thin fraction (also called the supernatant) by means of a centrifuge. A self-developed laboratory setup for ammonium removal and recovery is used for the treatment of the TAN-rich thin fraction. This setup is described in detail in Sect. "Setup for ammonium removal by stripping and scrubbing". The thin fraction, with a significantly reduced TAN level after stripping, is combined with a thick fraction obtained after centrifugation to reconstitute the whole digestate. After addition of substrate, the mixture is subjected to an anaerobic digestion and the result is compared with that of an untreated digestate from which no TAN was removed. In this way, it can be investigated whether TAN exerts a possible inhibitory effect during anaerobic digestion and consequently on the biogas production for this specific digestate. The setup used for carrying out the thermophilic biogas experiments is described in Sect. "Setup for carrying out the thermophilic biogas experiments"; the investigation of the inhibitory effect of TAN is discussed in Sect. "Investigation of the inhibitory effect of ammonium".

Origin of the digestate and substrate

To perform this laboratory-scale study, 25 L of substrate (received on April 18, 2016) and 50 L of digestate (received on August 8, 2016) were obtained from Mydibel (Mouscron, Belgium). Mydibel is an innovative Belgian family food business which produces both fresh, deep frozen and dried potato products. Mydibel attaches great importance to sustainability and ferments the starch-rich streams from the production at its own Green Factory site into biogas [25].

The digestate was from a thermophilic digestion (operating temperature of 323.15 K or 50 °C). The substrate is a suspension of biomass fed to the digester and comprises glazed potatoes, potato peels, rejected fried product, etc. Prior to characterization, both the substrate and digestate were blended to obtain a homogeneous mixture. In this way, the substrate is converted into heavily loaded organic wastewater. Afterwards, the substrate and digestate were characterized by analysing the following parameters: pH, chemical oxygen demand (COD), total organic carbon (TOC), ammonium, nitrite and nitrate.

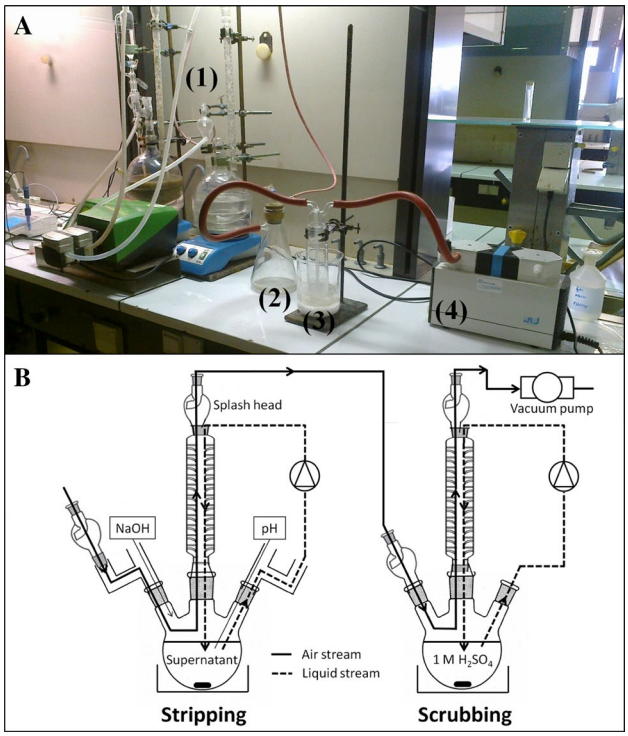

Fig. 2 **a** Picture of the complete self-developed laboratory-scale setup used for the ammonium removal and recovery and **b** schematic drawing of the stripping and scrubbing process

Setup for ammonium removal by stripping and scrubbing

In this laboratory study, it is investigated whether the ammonium present in the thermophilic digestate can be selectively removed and recovered via a stripping and scrubbing process, respectively. The self-developed laboratory-scale setup used for this ammonium removal and recovery is illustrated in Fig. 2.

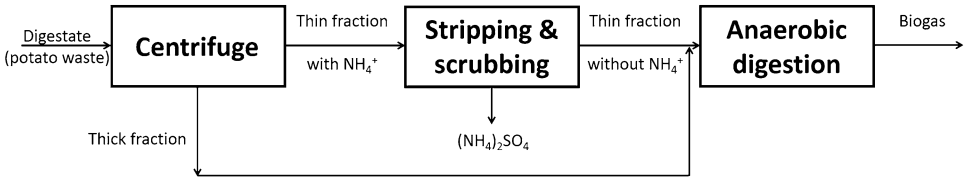

Fig. 1 Flowchart of the experimental procedure

Figure 2a shows the complete setup used for the ammonium removal in which four parts can be distinguished. With a laboratory vacuum pump (4) (KNF Laboport, USA), air is drawn through the stripping and scrubbing part of the setup equipped with Vigreux columns (total length of 0.4 m, 0.032 m diameter), a commonly used laboratory fractionating column (1). The Vigreux columns provide in both parts an exchange surface between the gas and the liquid phase that pass in counter-current through the columns. The vacuum is regulated by a valve (2) and a cold finger (3) is used to protect the vacuum pump against the gas vapours formed. The supernatant (in the stripping part) and the sulphuric acid solution (in the scrubbing part) were introduced into three-necked flasks of 5 and 3 L, respectively. Both Vigreux columns were provided with a splash head to prevent the supernatant and sulphuric acid solution ending up in the scrubbing part and valve/cold finger, respectively, because of the air stream through the entire system.

Figure 2b shows a schematic drawing of the stripping and scrubbing process. In the stripping part, the supernatant (thin fraction) of the digestate is continuously stirred using a magnetic stir bar and is continuously pumped over a Vigreux column at a flow rate of 7 L h^{-1} using a peristaltic pump (Watson–Marlow 520S, Belgium). The 2000 mL of supernatant used was obtained by centrifugation of 2450 mL digestate at 4000 rpm (Jouan C412, Thermo Fisher Scientific, USA). The ammonium present in the supernatant is volatized as ammonia through the combination of temperature increase and pH increase. As we did not want to deactivate the microorganisms present in this supernatant—as this could negatively influence further digestion—we were limited in both the temperature and pH increase that could be applied. We used a temperature of 323.15 K (50 °C), which was the temperature of the thermophilic reactor from which the original digestate was taken, and the pH was only increased from 7.8 to 8.5 using 4 M NaOH. The heating happened with a glass hot water bath and NaOH was added dropwise with an addition funnel of 0.25 L. During the stripping process, we aimed for a significant ammonium removal of 90%; the monitoring was done through regular sampling. The air that is drawn through the system takes the ammonia from the stripping part to the scrubbing part, which initially contains 1 L H_2SO_4 1 M. The sulphuric acid is also continuously stirred and pumped over a Vigreux column at a flow rate of 7 L.h^{-1} resulting in the formation of $(NH_4)_2SO_4$. The stirring (in both the stripping and scrubbing part) and heating (only in the stripping part) was accomplished by a Heating Magnetic Stirrer FB 15001 from Thermo Fisher Scientific (USA).

This procedure for ammonium removal allows us to investigate the effect of ammonium on the digestion process for this particular digestate. For this purpose, two biogas experiments were performed and compared. For the first experiment, the ammonium was removed from the supernatant (by means of stripping). After the ammonium removal, the pH of the supernatant was lowered to the original value (pH 7.8) with concentrated HCl and then was recombined with freshly centrifuged thick fraction. The digestion started after the addition of 50 mL of substrate to this mixture. The result of this experiment is compared to that of an experiment with untreated digestate in which the ammonium was not removed by stripping. For this also, 50 mL of substrate was added to the untreated digestate after which the digestion was started. Both experiments were performed at 323.15 K (50 °C) in the configuration described in 'Setup for carrying out the thermophilic biogas experiments'. Next, in an effort to optimize this process on laboratory scale, also another frequently used fractionating column was tested for its gas–liquid partition characteristics, namely a Dufton column (total length of 0.29 m, 0.029 m diameter). In a Dufton column, downwards spiralling liquid normally meets ascending vapour. In our application, a downwards spiralling H_2SO_4 solution meets the ascending ammoniac. Two additional combinations were tested: (1) a Dufton column in the stripping part and a Vigreux column in the scrubbing part and (2) vice versa. The two types of fractionating columns used in this study are schematically shown in Fig. 3.

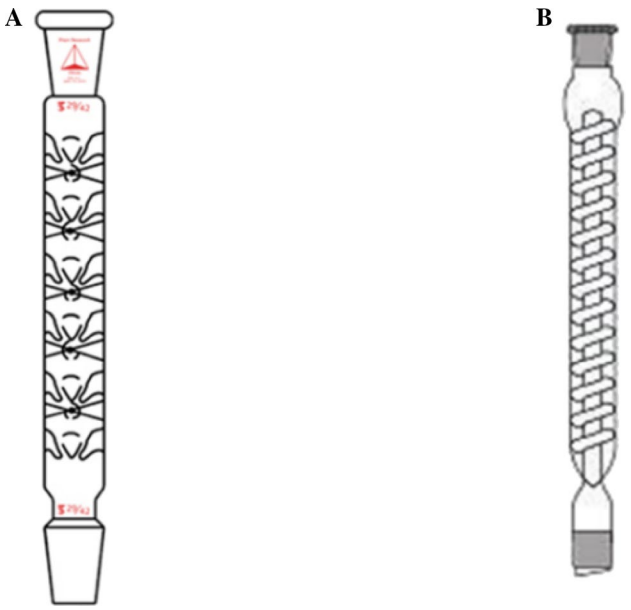

Fig. 3 Schematic representation of two types of commonly used laboratory fractionating columns, namely **a** the Vigreux column and **b** the Dufton column

Setup for carrying out the thermophilic biogas experiments

To investigate the possible inhibitory effect of ammonium in the thermophilic digestate of a potato processor, two experiments were carried out with (1) treated digestate in which the ammonium present in the digestate was removed via stripping and scrubbing and (2) untreated digestate in which the ammonium present was not removed. Both experiments were performed in the same setup used in our previous research as the basic configuration for the mesophilic biogas experiments [23]. Figure 4 shows the schematic drawing of this configuration.

The digestate (2450 mL) and the mixed substrate (50 mL) are introduced into an Erlenmeyer flask of 3 L (1). The digestate is thus either untreated digestate (no ammonium removal) or treated digestate (ammonium removal from supernatant + recombination with freshly obtained thick fraction after centrifugation). This flask is placed in an incubator (Memmert, Germany) with an operating temperature of 323.15 K or 50 °C (digestate was taken from a thermophilic digestion with the same operating temperature). The flask is connected with a recipient filled with 0.01 M H_2SO_4 (2) by means of a gas-tight tubing. A sulphuric acid solution is used to prevent the growth of algae. The volume of biogas formed is measured by the pressure-driven displacement of the sulphuric acid solution from recipient 2 to the graduated recipient 3. In this way, the biogas production can be accurately monitored to investigate the possible inhibitory effect of ammonium. The experiments are stopped when the pressure-driven displacement of sulphuric acid solution towards the graduated recipient more or less stagnated and the process thus is completed.

Analytical methods

For the characterization of both the digestate as the mixed substrate, the following parameters were analysed: pH, total organic carbon (TOC), chemical oxygen demand (COD), ammonium, nitrite and nitrate. The pH was measured with a S220 SevenCompact pH benchtop meter equipped with an InLab Expert Pro-ISM electrode (Mettler Toledo, Switzerland). The TOC concentration was measured with a TOC analyser (TOC-V_{CPN} Total Organic Carbon Analyzer, Shimadzu, Japan). COD was determined through the combination of the HI 839800 COD reactor and the HI 83214 Multiparameter Bench Photometer for Wastewater Treatment Application (Hanna Instruments, USA). The method using potassium dichromate is an adaptation of the USEPA 410.4-approved method for the COD determination on surface waters and wastewaters. Ion concentrations (ammonium, nitrite and nitrate) were measured by an ion chromatograph equipped with a conductivity detector (883 Basic IC Plus and 883 Compact Autosampler, Metrohm, Switzerland). For the detection of ammonium (in addition for the characterization, also for monitoring the ammonium removal during the stripping and scrubbing), the ion chromatograph was supplied by the combination of a Metrosep C-4—150/4.0 analytical column and Metrosep C-4 Guard/4.0 precolumn with 0.7 mM dipicolinic acid and 1.7 mM HNO_3 as eluent at a constant flow rate of 0.9 mL min^{-1}. The anions (nitrite and nitrate) were separated on a Metrosep A Supp 5 150/4.0 analytical column and Metrosep A Supp 4/5 Guard precolumn with 3.2 mM Na_2CO_3 and 1.0 mM $NaHCO_3$ as eluent at a constant flow rate of 0.7 mL min^{-1}.

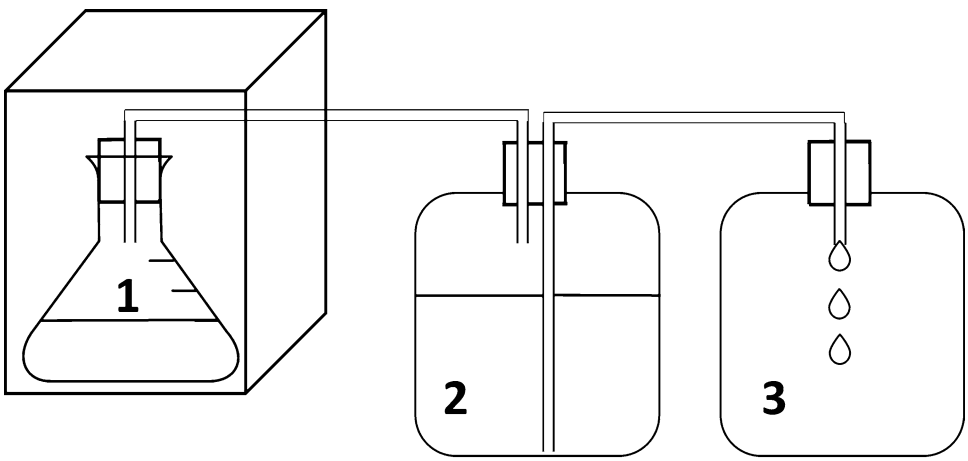

Fig. 4 Schematic drawing of the configuration used for carrying out two thermophilic biogas experiments to investigate the possible inhibitory effect of ammonium

Results and discussion

Characterization of the thermophilic digestate and substrate

The characteristics of the thermophilic digestate and substrate are given in Table 1. Prior to characterization, both the substrate and digestate were blended to obtain a homogeneous mixture.

The substrate has a high concentration of organic material as can be seen from the high TOC and COD value. The value of ammonium is quite low and nitrite/nitrate was zero (below the detection limit of the ion chromatograph). The analysis of the thermophilic digestate clearly shows that there is a significant reduction in the concentration of TOC and COD (compared to the substrate) due to the digestion process. This strong decrease (approximately, 90%) is due to the starch present in the substrate (derived from the potato products), which is very readily biodegradable. The residual concentration of organic material (approximately, 10%) is probably due to lignocellulosic structures present in the potato peels [26]. The conversion efficiency of this starch-rich substrate is thus significantly higher than the 50% that is mentioned in the introduction. In addition, the digestion process gives rise to a serious increase of ammonium which obviously is due to the release of the organically bound nitrogen. Moreover, Table 1 clearly shows that the digestate does not contain nitrite and nitrate.

Investigation of the inhibitory effect of ammonium

As can be seen from Table 1, the thermophilic digestate has a moderately high ammonium content of 2.3 g L^{-1} NH_4^+–N. The application of an oxidation technique to increase the biodegradability of the digestate can lead to the oxidation of the ammonium present to nitrite and/or nitrate. The latter nitrogenous components will then be denitrified in the subsequent digestion process with the loss of biogas production potential, as proven in our previous research [23]. In this context, it is examined whether it is possible to remove the ammonium from the digestate (more precisely, from the supernatant obtained after centrifugation of the thermophilic digestate) by means of a stripping and scrubbing process. The laboratory-scale setup used for this ammonium removal is illustrated in Fig. 2.

Table 1 Characteristics of the thermophilic digestate and substrate

	Substrate	Digestate
pH (–)	3.9	7.8
NH_4^+ (g L^{-1} N)	0.2	2.3
NO_2^- (g L^{-1} N)	0	0
NO_3^- (g L^{-1} N)	0	0
TOC (g L^{-1} C)	46	4.4
COD (g L^{-1} O_2)	212	40

Initially, the self-developed laboratory-scale setup was equipped with two Vigreux columns, which is a commonly used laboratory fractionating column: one for the stripping part and the other for the scrubbing part. During the stripping, the ammonium present in the supernatant is volatilized into ammonia through a combination of temperature and pH increase. As we did not want to deactivate the microorganisms present in this supernatant—as this could negatively influence the further digestion—we were limited in both the temperature and pH increase that could be applied. We used a temperature of 323.15 K (50 °C), which was the temperature of the thermophilic reactor from which the original digestate was taken, and the pH was only increased from 7.8 to 8.5. During the experiment, 89% of the ammonium present in the supernatant was removed; the final TAN concentration was 0.25 g L^{-1}. This stripping efficiency is quite similar to that obtained by Jiang et al. [20], although their experimental conditions of the stripping process were different from ours, namely the use of digested dairy manure, temperature of 308.15 K (35 °C) and pH of around 10. In our experiment, simultaneously 80% of the stripped ammonia was absorbed in 1 M H_2SO_4 in the scrubbing part. This results in the formation of $(NH_4)_2SO_4$. After stripping the ammonium, the pH of the treated supernatant was lowered to the original pH of 7.8 by means of concentrated HCl and recombined with freshly obtained thick fraction (obtained after centrifugation of digestate). After the addition of 50 mL of substrate the digestion was started. This experiment is referred to as the treated digestate. To examine the effect of the (reduction in) ammonium concentration, this experiment is compared to an untreated digestate in which the ammonium was not removed. Also in this case, 50 mL of substrate was added to the untreated digestate, after which the digestion was started. Both experiments were performed at 323.15 K (50 °C) in the configuration shown in Fig. 4. Figure 5 shows the volume of biogas produced for both single experiments.

Figure 5 shows that the volume of biogas produced is similar for both the treated and untreated digestate during the first 4 days of both experiments. From that point, it is noted that both profiles start to diverge. After about 8 days, it is observed that the biogas yield is 36% higher if the ammonium was removed from the digestate (treated digestate) in comparison to the untreated digestate. Furthermore, a control experiment (single experiment) was performed to investigate the effect of the centrifugation step on biogas production. Figure 5 clearly shows that the progress in biogas production of the untreated digestate and the control experiment almost coincide. Despite the

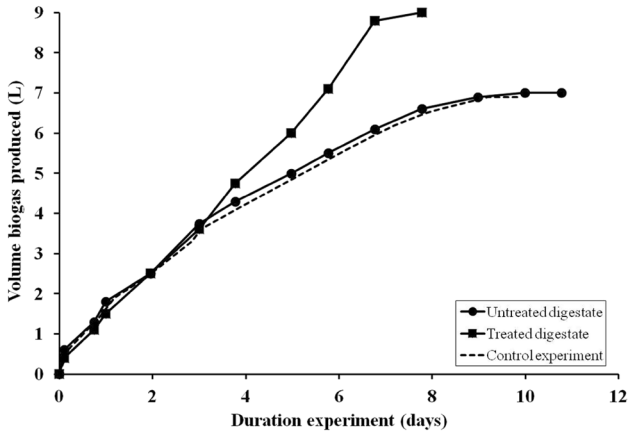

Fig. 5 Volume of biogas produced for both the treated (TAN content of 0.25 g L^{-1} after stripping) and untreated digestate (TAN content of 2.3 g L^{-1}). Control experiment was performed to investigate the effect of the centrifugation step on the biogas production (TAN content of 2.3 g L^{-1})

difference in performance, namely that the untreated digestate was not subjected to a centrifugation step in contrast to the control experiment, we can state that the experiments are repeatable. This proves that (1) the centrifugation of the digestate into a thick fraction and a supernatant does not affect the biogas formation as a digestion experiment on the combined fractions resulted in the same biogas production as the untreated digestate and (2) the higher biogas yield using the treated digestate is therefore not due to the centrifugation step but due to the reduced TAN level.

As the pH of the incubation mixtures (digestate + substrate) remained constant at about pH 7.8 during the entire digestion process, there was no effect on the equilibrium of NH_4^+ and NH_3. As the pK_a of the NH_4^+/NH_3 system is 9.2, it is obvious that most of the TAN occurs as NH_4^+. This implies that the inhibitory effect in the untreated digestate probably is due to NH_4^+–N. The total TAN concentration of 2.3 g L^{-1} (see Table 1) is within the range of 1.7–6.7 g L^{-1} TAN, and it is known that it may exert an inhibitory effect on the methanogenesis step [10, 11].

To avoid ammonium inhibition and thus to ensure a higher biogas yield, it is therefore appropriate to remove the ammonium prior to the digestion process. The stripping and scrubbing process provides an efficient way to achieve this removal and recovery. The related benefits of implementing the stripping and scrubbing process are threefold: (1) after the application of an advanced oxidation process (AOP), denitrification of nitrite/nitrate will not occur because these nitrogenous components cannot be formed anymore or at least in much lower concentrations; (2) reduction of the ammonium content in the digestate is favourable in view of the inhibitory effect of this component and (3) the removed ammonium is recovered as $(NH_4)_2SO_4$, which can be used as an N–S fertilizer.

Optimization of the stripping and scrubbing process on laboratory scale

As described above, two Vigreux columns were used in the first stripping and scrubbing experiment for gas–liquid exchange. In an effort to optimize to some extent this gas–liquid exchange in our particular conditions which limits the temperature and pH that can be used (since the microorganisms present must stay viable for the subsequent digestion experiment), also another frequently used laboratory fractionating column was used for gas–liquid exchange, namely a Dufton column (see Fig. 3b). Two additional column combinations were studied: (1) a Dufton column in the stripping part and a Vigreux column in the scrubbing part and (2) vice versa. The three combinations were tested once and were assessed based on three criteria. The first criterion is the percentage ammonium removal from the supernatant. The second criterion is the time interval required for this removal. The third criterion is the recovery of the removed NH_4^+ in 1 M H_2SO_4. The first two criteria mentioned are related to the stripping part; the latter is related to the scrubbing part. Figure 6 gives an assessment of the stripping and scrubbing process for the three column combinations investigated.

The column combination Dufton (stripping part)–Vigreux (scrubbing part) allows to recover 96% of the stripped ammonium during the scrubbing phase. However, this is the most unfavourable column combination because the other two criteria were far from optimal, namely a low ammonium removal percentage (67%) in combination with the largest time interval (28 h). The other two column combinations are more in balance in terms of results on the stripping and scrubbing part. Although a larger time interval is required to realize a similar percentage of ammonia removal (about 90%), there is a slight preference for the column combination Vigreux (stripping)–Dufton (scrubbing) since a larger percentage of the stripped ammonium is recovered.

Even in the best combination, the time interval that was necessary for stripping was 17 h, which is quite long. We, however, have to take into consideration that only a limited increase of temperature (323.15 K or 50 °C) and pH (8.5) was applied, as we did not want to deactivate the microorganisms that were present in the supernatant of the digestate. The system itself was not optimized for the stripping step, but it is obvious that the process would benefit from increasing working temperature and pH. In our self-developed laboratory setup, fractionating columns (in particular, Vigreux columns) were used to investigate ammonium removal and to demonstrate the resulting decrease of the inhibitory effect of NH_4^+ during the subsequent digestion

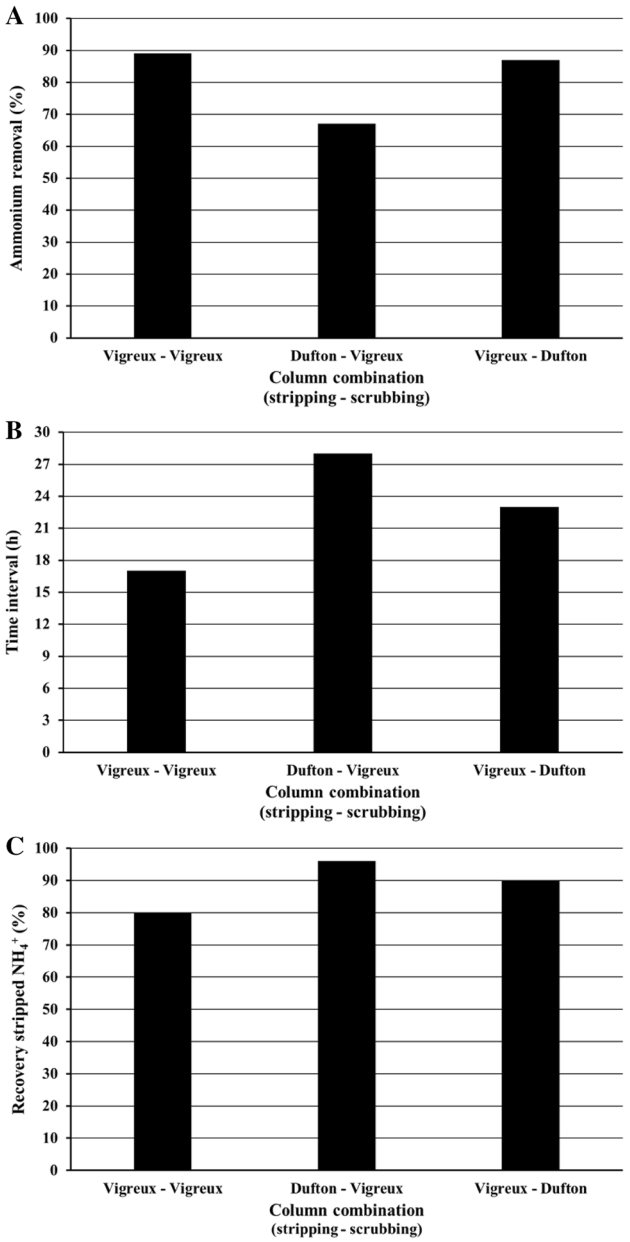

Fig. 6 Assessment of the stripping and scrubbing process for the three column combinations investigated. **a** Percentage ammonium removal from the supernatant during stripping; **b** time interval required for this ammonium removal; **c** recovery of the stripped NH_4^+ in 1 M H_2SO_4 during scrubbing

process. However, we believe that scaling up of the results obtained (significant ammonium removal and recovery) towards pilot scale or even full scale is certainly realistic. For these scale sizes, the use of packed columns will be more appropriate instead of the commonly used laboratory fractionating columns [17].

In this laboratory study, the ammonium removed from the supernatant (digestate) is recovered as $(NH_4)_2SO_4$, which can act as a nitrogen fertilizer. In principle, it is also possible to use HNO_3 (instead of H_2SO_4) in the scrubbing part. Absorption of the stripped NH_4^+ results in this case in NH_4NO_3, which has a higher intrinsic value as nitrogen fertilizer.

Conclusion

Anaerobic digestion is generally considered as one of the most sustainable technologies for the production of renewable energy. The main bottleneck of the digestion process is the rather low conversion efficiency of organic material which is partly due to the accumulation of ammonia/ammonium. This nitrogen issue can thus be avoided by removing the ammonium present in the digestate by means of a stripping and scrubbing process. In this study, a self-developed laboratory-scale setup for stripping and scrubbing was developed and applied on a thermophilic digestate from a potato processor. This easily built system can be used to study stripping and scrubbing of any digestate. Our digestion experiments have demonstrated that after 8 days of incubation, the biogas yield is 36% higher after removal of the ammonium from the digestate compared to the untreated digestate. This proves the inhibitory effect of ammonium at a concentration of 2.3 g L^{-1} NH_4^+–N for this particular digestate. In addition, the stripping and scrubbing process ensures that ammonium is removed from the digestate and recovered in the form of $(NH_4)_2SO_4$, which can act as nitrogen fertilizer. The stripping and scrubbing process was investigated with three different combinations of two types of commonly used laboratory fractionating columns, namely a Vigreux column and a Dufton column for their gas–liquid partition potential. Without optimizing for ammonium removal, the column combination Vigreux (for the stripping part)—Dufton (for the scrubbing part) could remove 87% of the ammonium from the supernatant (obtained after centrifugation of the digestate), while simultaneously 90% of the stripped ammonium was recovered as $(NH_4)_2SO_4$. The overall efficiency for the nitrogen recovery is thus almost 80%.

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Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

References

1. Jingura, R.M., Matengaifa, R.: Optimization of biogas production by anaerobic digestion for sustainable energy development in Zimbabwe. Renew. Sustain. Energ. Rev. **13**, 1116–1120 (2009)
2. Lee, M., Hidaka, T., Hagiwara, W., Tsuno, H.: Comparative performance and microbial diversity of hyperthermophilic and thermophilic co-digestion of kitchen garbage and excess sludge. Bioresour. Technol. **100**, 578–585 (2009)
3. Khalid, A., Arshad, M., Anjum, M., Mahmood, T., Dawson, L.: The anaerobic digestion of solid organic waste. Waste Manage. **31**, 1737–1744 (2011)
4. Achinas, S., Achinas, V., Euverink, G.J.W.: A technological overview of biogas production from biowaste. Engineering **3**, 299–307 (2017)
5. Matsakas, L., Gao, Q., Jansson, S., Rova, U., Christakopoulos, P.: Green conversion of municipal solid wastes into fuels and chemicals. Electron. J. Biotech. **26**, 69–83 (2017)
6. Schievano, A., Tenca, A., Lonati, S., Manzini, E., Adani, F.: Can two-stage instead of one-stage anaerobic digestión really increase energy recovery from biomass? Appl. Energ. **124**, 335–342 (2014)
7. Goswami, R., Chattopadhyay, P., Shome, A., Banerjee, S.N., Chakraborty, A.K., Mathew, A.K., Chaudhury, S.: An overview of physico-chemical mechanisms of biogas production by microbial communities: a step towards sustainable management. 3. Biotech **6**(72), 1–12 (2016)
8. Park, C., Lee, C., Kim, S., Chen, Y., Chase, H.A.: Upgrading of anaerobic digestion by incorporating two different hydrolysis processes. J. Biosci. Bioeng. **100**, 164–167 (2005)
9. Mumme, J., Linke, B., Tölle, R.: Novel upflow anaerobic solid-state (UASS) reactor. Bioresour. Technol. **101**, 592–599 (2010)
10. Yenigün, O., Demirel, B.: Ammonia inhibition in anaerobic digestion: a review. Process Biochem. **48**, 901–911 (2013)
11. Franke-Whittle, I.H., Walter, A., Ebner, C., Insam, H.: Investigation into the effect of high concentrations of volatile fatty acids in anaerobic digestion on methanogenic communities. Waste Manage. **34**, 2080–2089 (2014)
12. Magrí, A., Béline, F., Dabert, P.: Feasibility and interest of the anammox process as treatment alternative for anaerobic digester supernatants in manure processing—an overview. J. Environ. Manage. **131**, 170–184 (2013)
13. Escudero, A., Blanco, F., Lacalle, A., Pinto, M.: Struvite precipitation for ammonium removal from anaerobically treated effluents. J. Environ. Chem. Eng. **3**, 413–419 (2015)
14. Pastor, L., Mangin, D., Ferrer, J., Seco, A.: Struvite formation from the supernatants of an anaerobic digestion pilot plant. Bioresour. Technol. **101**, 118–125 (2010)
15. Bonmatí, A., Flotats, X.: Air stripping of ammonia from pig slurry: characterisation and feasibility as a pre- or post-treatment to mesophilic anaerobic digestion. Waste Manag. **23**, 261–272 (2003)
16. Laureni, M., Palatsi, J., Llovera, M., Bonmatí, A.: Influence of pig slurry characteristics on ammonia stripping efficiencies and quality of the recovered ammonium-sulfate solution. J. Chem. Technol. Biotechnol. **88**, 1654–1662 (2013)
17. Srinath, E.G., Loehr, R.C.: Ammonia desorption by diffused aeration. Water Pollut. Control Fed. **46**, 1939–1957 (1974)
18. Limoli, A., Langone, M., Andreottola, G.: Ammonia removal from raw manure digestate by means of a turbulent mixing stripping process. J. Environ. Manage. **176**, 1–10 (2016)
19. Provolo, G., Perazzolo, F., Mattachini, G., Finzi, A., Naldi, E., Riva, E.: Nitrogen removal from digested slurries using a simplified ammonia stripping technique. Waste Manag. **69**, 154–161 (2017)
20. Jiang, A., Zhang, T., Zhao, Q.-B., Li, X., Chen, S., Frear, C.S.: Evaluation of an integrated ammonia stripping, recovery, and biogas scrubbing system for use with anaerobically digested dairy manure. Biosyst. Eng. **119**, 117–126 (2014)
21. Yuan, M.-H., Chen, Y.-H., Tsai, J.-Y., Chang, C.-Y.: Removal of ammonia from wastewater by air stripping process in laboratory and pilot scales using a rotating packed bed at ambient temperature. J. Taiwan Inst. Chem. E. **60**, 488–495 (2016)
22. Bousek, J., Scroccaro, D., Sima, J., Weissenbacher, N., Fuchs, W.: Influence of the gas composition on the efficiency of ammonia stripping of biogas digestate. Bioresour. Technol. **203**, 259–266 (2016)
23. Ghyselbrecht, K., Monballiu, A., Somers, M.H., Sigurnjak, I., Meers, E., Appels, L., Meesschaert, B.: The fate of nitrite and nitrate during anaerobic digestion. Environ. Technol. (2017). https://doi.org/10.1080/09593330.2017.1415380
24. Zarebska, A., Romero Nieto, D., Christensen, K.V., Fjerbæk, Søtoft L., Norddal, B.: Ammonium fertilizers production from manure: a critical review. Crit. Rev. Environ. Sci. Technol. **45**, 1469–1521 (2014)
25. Mylle, C.: The Mydibel sustainability report (September 2016). http://www.mydibel.be/sites/default/files/files/basic_pages/files/mydibel_sustainability_report_lr.pdf. Accessed 08 February 2017
26. Ge, X., Xu, F., Li, Y.: Solid-state anaerobic digestion of lignocellulosic biomass: recent progress and perspectives. Bioresour. Technol. **205**, 239–245 (2016)