EFFECTIVE MANAGEMENT OF SEWAGE SLUDGE

(NH₄)₂SO₄ recovery from liquid side streams

Marc Anton Boehler • Alexander Heisele • Alexander Seyfried • Markus Grömping • Hansruedi Siegrist

Received: 20 March 2014 / Accepted: 28 July 2014 / Published online: 19 August 2014 © Springer-Verlag Berlin Heidelberg 2014

Abstract Two methods of recovering nitrogen from liquid side streams are presented in this paper. The first method was demonstrated at an ammonia stripping plant treating $5-7 \text{ m}^3/\text{h}$ sludge water at the wastewater treatment plant (WWTP) Kloten-Opfikon (CH). In addition to the usual stripping and scrubbing columns, a third column had been added in order strip CO₂, thus reducing the NaOH-demand of the subsequent ammonia stripping. At first, just the stripping plant was put into operation and optimized without any pre-treatment of the supernatant. Next, the CO2-stripper column was activated and optimized by gas measurements to minimize free ammonia losses, heat losses, and energy consumption. Key operational aspects of the plant were evaluated. Finally, up to $1.4 \text{ m}^3/\text{h}$ source-separated urine was successfully fed into the stripping facility. The second ammonia removal method using hydrophobic hollow fiber membranes was tested in two small pilot systems by different manufacturers in 2012 and 2013 at WWTP Neugut. In this technology, free ammonia gas in the sludge liquid diffuses at pH >9.3 from the sludge liquid through the air-filled pores of the microporous hydrophobic membrane into concentrated sulfuric acid flowing through the hollow fibers, forming ammonium sulfate. The small pore size and the hydrophobic nature of the membrane prevent the liquid phase from entering into the pores due to the surface tension effect. Practical experience regarding operational parameters like wastewater flow rate, pH, temperature, ammonia

Responsible editor: Bingcai Pan

M. A. Boehler (\boxtimes) · A. Heisele · H. Siegrist Eawag: Swiss Federal Institute of Aquatic Science and Technology, Process Engineering, Überlandstrasse 133, Postfach 611, 8600 Dübendorf, Switzerland e-mail: marc.boehler@eawag.ch

A. Seyfried · M. Grömping ATEMIS GmbH, Aachen, Germany concentration, fouling and precipitations processes, optimal flow schemes, and process configurations was collected.

Keywords Nutrient recovery \cdot Ammonia stripping \cdot Air stripping \cdot Membrane stripping \cdot Membrane contactor \cdot Fertilizer \cdot Side streams

Background and motivation

In the last 50 years, the production of artificial fertilizer surged, increasingly loading both water bodies and the atmosphere with nitrogen-compounds. Today there are many efforts to break this trend, as for example the EU directive 91/676/EWG of the European Community from 1991 which aims to protect water bodies and to reduce nitrate emissions.

The production of nitrogen fertilizer via the Haber-Bosch process and the elimination of the nitrogen from municipal waste water streams by denitrification are cost and energy intensive. The latter also produces nitrous oxide (N_2O), a very strong greenhouse gas. In this context, technologies for nutrient separation and recovery out of the waste water will get more and more attention in the coming future.

Novel ammonia air stripping process at WWTP Kloten/Opfikon

Free ammonia air stripping requires large amounts of energy intensive and costly chemicals like sodium hydroxide and sulfuric acid to produce ammonium sulfate. Costs and energy consumption to produce the NaOH are a significant part of the overall operational costs and energy consumption of an air stripping plant.

Only about 10 pre-treatment plants in Europe are using the air stripping process and produce fertilizer in the form of

ammonium sulfate. Some of the stripping plants were designed by the ROUTES project partner and engineering company ATEMIS (Germany). These full scale plants are located at wastewater treatment plants (WWTPs) e.g., in Straubing und Wallau (Germany), as well as in Spittal (Austria).

The novel process—implemented in full scale and continuously operated at the WWTP Kloten/Opfikon since spring 2011—uses optimized pH and temperature conditions for an average nitrogen elimination of about 90 %. This air stripping plant for the recovery of nitrogen is the first one in Switzerland (Boehler M. Liebi C 2012).

The air stripping plant is installed in the former sludge drying hall of WWTP Kloten/Opfikon. In addition to a conventional air stripping plant consisting of a NH₃ stripper and an acidic scrubber column, it was provided with a second stripping column for the pre-treatment of the sludge water by stripping CO₂ to reduce the H₂CO₃ concentration in the sludge liquid. This pre-treatment was expected to raise the pH of the sludge liquid and to reduce the NaOH required to increase the pH further. Figure 1 shows the flow scheme of the stripping plant at the WWTP Kloten-Opfikon (CH).

In a very first step, the sludge liquid is filtered with a disk filter to remove small organic and plastic solids (100 μ m filter). The efficient removal of solids is of importance regarding the optimal operation of the columns (see further explanation in section "Results air stripping"). In a further step, the sludge liquid is heated up to 60 °C by a heat exchanger and then pumped to the top of the CO₂-stripping column. Source-separated urine can be treated together with the sludge liquid in the air stripping plant.

Figure 2 shows the view and bottom part of the plant. The whole stripping plant is about 11 m high and spans three floors. The columns are well isolated to prevent heat losses. The CO₂-stripper is operated with an off-gas stream, whereas the free ammonia stripper and acidic scrubber are operated with a recycling gas stream. The process gas is recycled from the scrubber to the stripper (gas loop).

At the top of the CO₂ and of the NH₃-columns, the sludge water is distributed over the whole cross section via feed trays. The columns are packed with support media to increase the water/air interface. In the stripper columns, the air and sludge liquid flows are in opposite directions in order to maximize the stripping efficiency. Carbon dioxide is about one thousand times more volatile than NH₃. It is stripped in the first column with a small air flow rate without losing significant amounts of NH₃ (approx. 2.5%) with the produced off-gas. In this process step, the pH value of the sludge water phase is raised, which helps to reduce the amount of base required to shift the NH₃/ NH₄-ratio towards NH₃. Next, the sludge water is pumped to the top of the NH₃-stripper. Before reaching the NH₃-stripper, the pH is adjusted to a defined value by dosing sodium hydroxide. As with the CO₂-stripper, the support media helps to increase the water/air interface. In contrast to the CO₂-

stripper, a large amount of air is brought in contact with the sludge water to transfer the low volatile free ammonia from the liquid phase to the gas phase. In order to maximize the stripping efficiency, air and sludge liquid flow in opposite directions.

In the acid scrubber, the free ammonia of the gas phase is brought in contact with highly concentrated sulfuric acid. Due to the low pH value of the acid, all free ammonia is converted to NH₄ and an ammonium sulfate solution with about 10 % ammonia (corresponding to about 100 g N/L) and a pH value of approximately 4.5 to 5 (see Eq. 1) is produced. The gas of the scrubber column is recycled back in a gas loop (see Fig. 1) to the stripping column to prevent heat loss.

$$2 \text{ NH}_3 + \text{H}_2 \text{SO}_4 \rightarrow (\text{NH}_4)_2 \text{SO}_4 \tag{1}$$

In the first year of operation, the conventional free ammonia stripping as well as the CO_2 pre-stripping was optimized by Eawag and ATEMIS GmbH (Boehler et al. 2013b). At first, the new stripping plant was put into operation without any pre-treatment of the sludge liquid and was optimized in terms of optimal temperature, optimal pH, and optimal air demand of the NH₃-stripper (air/sludge liquid flow). Then, the plant was optimized in terms of minimization of base (NaOH) consumption by CO_2 pre-stripping, minimization of air demand for the CO_2 -stripper under respect of heat and NH₃-losses (off-gas) and optimal CO_2 stripping, quality of the product ammonium sulfate, the feasibility and efficiency of a co-treatment of source-separated urine to increase the amount of nitrogen recovered, and to reduce specific process costs and energy.

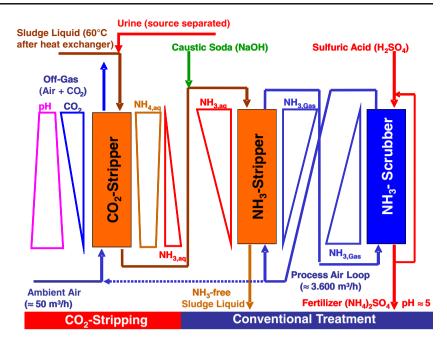
Beside the above listed key aspects, operational questions like pre-treatment of the sludge water (filtration), cleaning of the columns (acid cleaning), optimal support media, etc. were investigated during the project.

Results air stripping

The new stripping plant was put into operation without any pre-treatment of the sludge liquid. In the first year of operation, the conventional free ammonia stripping as well as the CO_2 pre-stripping was optimized by Eawag and ATEMIS GmbH. In a first step, the free ammonia stripping column was optimized in terms of temperature, pH, and optimal air demand (air/sludge liquid flow). As a result of tests with various settings, it was decided to operate the plant with the following operational key parameters: a temperature of the sludge liquid of 60 °C, a pH value of 9.3, a sludge liquid flow of 5.25 m³/h, and an air flow of 3,600 Nm³/h (resulting in an air/liquid ratio of 685).

In summer 2011, the co-treatment of sludge liquid with source-separated urine (up to 10% of the treated sludge water,

Fig. 1 Flow scheme of free ammonia air stripping with pretreatment by CO₂ stripping and optional co-treatment of sourceseparated urine



corresponding to 500 L/h; total treated urine about 2.7 m³) was tested in full scale for the first time (Boehler et al. 2013a). Urine represents a high loaded nutrient solution with nitrogen concentration in the range of 1,300 up to 8,000 mg N/L depending on collection (i.e., dilution) and storage (i.e., losses by ammonia volatilization) (Morales et al. 2013; Udert et al. 2006). The urine was collected in the office buildings of Eawag, which are equipped with facilities for urine separation and storage. To avoid

clogging of the support media in the stripping columns, urine has to be pre-treated by phosphorous precipitation and filtration (Morales et al. 2013). Limiting magnesium oxide was added to complete struvite precipitation. The urine storage tank in the basement of the Eawag office building was used as precipitation tank. After dosing magnesium oxide and stirring the tank for 30 min, the separated urine was pumped through filter bags with a pore size of 100 μ m to remove the formed crystals and other



Fig. 2 Full scale air stripping plant at WWTP Kloten/Opfikon

solids from the urine. The total amount of treated urine in 2011 and 2012 was about 11 m^3 .

In this first urine co-treatment experiments, the overall elimination efficiency was reduced to 70 %. No strong foaming was observed. The reason for the reduced elimination was a limitation of base, because the base dosage unit at the inlet of the stripping reactor was not working correctly probably due to a drift of the pH-sensor. This was observed due to the low pH value of about 8 in the effluent of the stripper, indicating a limitation of free ammonia production.

In 2012, the full scale urine co-treatment trials were repeated by adding of 10 and 20 vol.% of source-separated urine (700 and 1,400 L/h; total amount of treated urine, 8.4 m³). This augmentation resulted in an increase of the ammonia concentration of the sludge liquid by about 35 and 80 %, respectively. The pH in the inlet of the stripping reactor was now controlled at a value of 9.3 to 9.5 with NaOH dosage. High nitrogen elimination efficiencies of approximately 95 % were observed. These very successful experiments demonstrated the feasibility of co-treatment of source-separated urine with sludge digester liquid.

Operational experiences

The pre-treatment of the sludge water is of importance for an efficient operation of the stripping plant.

Organic solids in the sludge water can easily clog the support media. In the first period of operation of the stripping plant at WWTP Kloten/Opfikon, huge amounts of organic solids were brought to the columns. The operation of the dewatering facilities (centrifuges) was not optimal. During start up and shut down of the centrifuges, the suspended solids in the sludge liquid were strongly increased and clogged the support media. This fouling layer could not be removed by acid cleaning. The accumulation of organic solids in the support media resulted in a strong increase of the specific weight of the packing material and caused a collapse of it. As a consequence, it was decided to change the support media of the NH₃-stripper. It was replaced with a new support media with less surface area (125 m^2/m^3) than the original one $(240 \text{ m}^2/\text{m}^3)$ in order to have less clogging and better cleaning properties. Additionally, a disk filter was installed to retain small plastics pieces, fibers, and hairs. The filter is back washed every 20 to 30 min depending on the hydraulic pressure drop.

Precipitates in the reactors, especially on the support media, are a serious problem for the operation of the stripping plant. Precipitates increase the hydraulic pressure drop of the columns which resulted in a higher energy demand for the blower and decrease the efficiency of the heat exchanger.

If there is no efficient dissolution of the precipitates, the precipitation products can cause a total blockage of the support media. This happened after the first year of operation. Due to the clogging of the packing material by organic solids and strong chemical precipitation, the whole support media of the CO_2 -stripper was totally blocked and had to be exchanged. Also in the second year of operation, a fraction of the support media was removed in both columns because of inefficient acid cleaning. Different acid cleaning strategies were tested. Best experiences were made with a manual acid cleaning. A batch of hydrochloric acid was recycled in the columns until a pH value of about 5 was reached in the recycled acid solution. Then, the acid was discharged and a second or third batch was added until there is no increase of the pH value in the cleaning acid. Experiences of the operators showed that this cleaning procedure with hydrochloric acid is very effective for the NH₃-stripper but less efficient for the CO_2 -stripper.

To better understand the precipitation mechanisms, the precipitates of both columns were taken and analyzed. The elemental composition of the precipitates were measured by X-ray fluorescence (XRF) and then identified by X-ray diffraction (XRD). The following precipitates were found: apatite (Ca₅[OH|(PO₄)₃]), brushite (CaHPO₄×2H₂O), and calcite (CaCO₃). In the CO₂-stripper, apatite and brushite were both found at the head of the column. At the bottom (drip pan), mainly apatite was detected and in general less calcite. In the NH₃-stripper, the precipitation product was mainly calcite, no appetite and brushite was found.

Apatite and brushite are minerals based on phosphorous. In the sludge liquid of WWTP Kloten/Opfikon, phosphorous is found in concentrations in the range of 5 to 10 g PO₄-P/ $m^{3}_{digester supernatant}$. Brushite and apatite are less soluble than calcite and are therefore precipitating first during the slight pH increase in the CO₂-stripper, whereas calcite is precipitating in the NH₃ stripper after the pH increase due to the added NaOH.

In the following, solubility test with different acids (hydrochloric acid, sulfuric acid, and nitric acid) were undertaken. Apatite is only slowly soluble with hydrochlorid acid whereas nitric acid showed a good dissolution of brushite and apatite. Therefore, the use of nitric acid for the acid cleaning of the CO_2 -stripper would be an option.

To reduce the precipitation of apatite and brushite, the operator considers dosing a small amount of $iron^{3+}$ into the sludge liquid storage tank to reduce the orthophosphate concentration.

However, mixing digester supernatant with stored urine can result in precipitation of struvite and calcium phosphate: urine has a high pH value and a high phosphate concentration, while the digester supernatant delivers the calcium and magnesium ions necessary for precipitation. To prevent scaling of the support media, the phosphate in the urine has to be precipitated in an upstream reactor by adding magnesium.

In the second year of operation, the sludge liquid flow was increased to about 7 m³/h, which resulted in a better distribution of the sludge liquid over the cross section of the column (in the first year the flow was kept at 5.25 m^3 /h). It was also

observed that the original feed trays on the top of the columns were badly designed and resulted in a poor distribution of the sludge liquid. Only four apertures in the feed tray distributed the water over the support media. This led beside a poor distribution also to an intense local precipitation and clogging of the support media and only local flushing. New feed trays were installed to optimize the distribution.

Quality of the product

Ammonium sulfate is a valuable fertilizer which can be used for liquid fertilization in agriculture. Especially, the CULTAN fertilization (Controlled uptake and long term ammonium fertilization) features some interesting advantages in comparison to conventional fertilization with granulate. Due to this, the product ammonium sulfate is very in demand.

The ammonium sulfate concentration is limited to 40 % salt content. Higher concentration of ammonium sulfate resulted in a crystallization of the product. In this form, the ammonium concentration is about 100 g N/L. In addition, the fertilizer solution contains about 8 % sulfur.

To reach this optimal composition of the product, it is of great importance to use concentrated sulfuric acid (>60 %) that is diluted by water vapor diffusing from the sludge liquid to the product solution due to the difference in the osmotic pressure. For the air stripping process, a concentration of the sulfuric acid of 75 % is of advantage to reach the optimal concentration of the product ammonium sulfate.

Besides the content of nutrients in the product, the absence of problematic substances is of importance. The product and sludge liquid of WWTP Kloten/Opfikon was analyzed for micro pollutants (MPs) by the ROUTES partner BfG (Bundesanstalt für Gewässerkunde, Koblenz, Germany). Three samples of the ammonium sulfate solution from different experimental periods were analyzed for MPs).

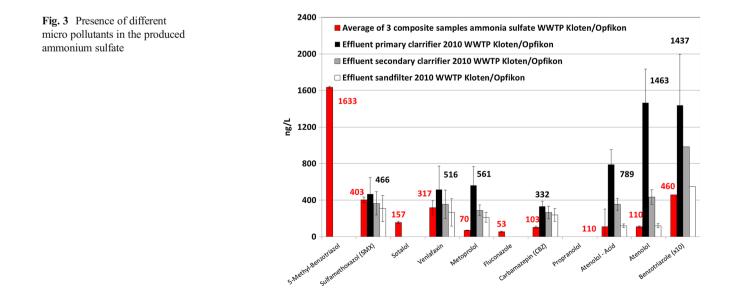
Figure 3 shows the results of the analysis for micro pollutants. Ten out of 28 analyzed MPs were found in the ammonium sulfate product, but in low concentrations compared to the primary effluent of the WWTP. Only about 0.05 ‰ of MP influent load of WWTP was found in the product. The micropollutant load to agriculture is therefore negligible compared to fertilization with sewage sludge.

Treatment costs and energy consumption

Beside optimization and testing of the full scale stripping plant of WWTP Kloten/Opfikon, a full scale cost calculation was done by the project partner ATEMIS, GmbH. On one hand, a detailed comparison of the expected treatment costs for a stripping plant with and without a CO₂-stripper showed only a small advantage of the CO₂-stripper (5.93 CHF vs. 5.85 CHF per kg N removed). On the other hand, the NaOHconsumption was reduced by about 40 to 50 %, resulting in considerably lower total energy consumption (11.98 vs. 9.67 kWh/kg N_{removed} for plant operation and chemicals). Since the energy costs can be expected to rise in the future, the cost advantage of a design with a CO₂-stripper can be expected to increase even further.

Design parameters for an NH₃ air stripping plant

The following table summarizes the evaluated processes and design parameters for the recovery of ammonia by air stripping and a pre-treatment by CO₂-stripping reducing the overall base consumption. All values are approximate values observed during the experiments. The values for the specific



🖄 Springer

Table 1 Key process and design parameters

Parameter	Value	Unit
Inflow temperature of the stripping plant	60	°C
Air/liquid ratio of the CO2-stripping column	8-10	_
Air/liquid ratio of the NH3-stripping column	700	_
Nitrogen removal efficiency	90 %	_
pH at the inflow of the NH3-stripping column	9.3-9.5	_
NaOH-demand w/o CO2-stripping	5.5-6.5	L NaOH (50 %)/kg N _{removed}
NaOH-demand with CO ₂ -stripping	3.5-4.0	L NaOH (50 %)/kg N _{removed}
Primary energy for electricity demand (approx. 1.4 kWh/kg N _{recovered} including 15 % for the CO ₂ -stripper)	4	kWh/kg N _{recovered}
Primary energy demand for chemicals (NaOH, H ₂ SO ₄) with CO ₂ stripper	6	kWh/kg N _{recovered}
Thermal energy demand (temp. diff. inlet/outlet, heat loss through walls and water evaporation in CO ₂ -stripper (8–9 °C=10 kWh/m ³) minus excess heat from biogas use (60 %))	4	kWh/kg N _{recovered}
Gross primary energy demand	14	kWh/kg N _{recovered}
Haber-Bosch process energy demand	-8	kWh/kg N _{produced}
Net primary energy demand	6	kWh/kg Nrecovered

base demand refer to 50 % NaOH solution. If there is thermal heat surplus from the WWTP, the net energy demand diminishes nearly to zero (Table 1).

Application of a half scale membrane module

Removal of ammonia using hydrophobic hollow fiber membranes has been tested in small pilot systems in 2012 and 2013 at WWTP Neugut. There is currently very limited data available to design full scale systems. Operating parameters such as waste water flow rate, pH, temperature, ammonia concentration as well as fouling and precipitations processes will significantly impact the ammonia removal characteristics and removal efficiency. Therefore, gathering more experience and experimental data is an important step towards developing and demonstrating this technology.

With this technology, only free ammonia can be removed from the sludge liquid. Free ammonia gas diffuses at high pH values from the sludge water across the airfilled pores of the microporous hydrophobic membrane as long as a sufficient driving force is maintained (Ulbricht et al. 2009). The small pore size and the hydrophobic nature of the membrane prevent the liquid phase from entering into the pores or flowing through the porous wall due to the surface tension effect. Because of the very low Henry constant and high solubility of NH₃ compared to other dissolved gases in water (e.g., CO_2 or O_2), the free ammonia gas will be difficult to remove by applying a vacuum or sweep gas-vacuum combination as in typical degassing operations with a membrane contactor technology. However, an acid will work very effectively in dissolving and removing the ammonia gas from the waste water. The low-pH sulfuric acid solution will instantly react with ammonia gas according to Eq. 1 to form ammonium sulfate. This will generate and maintain the

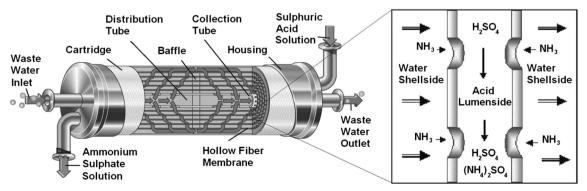
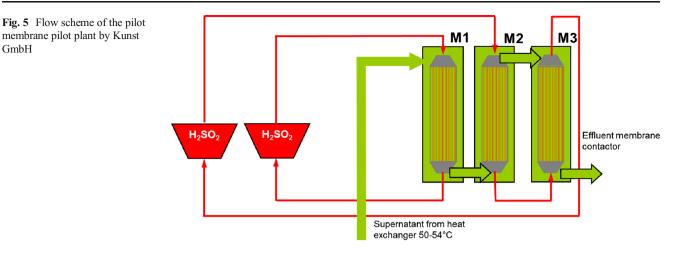


Fig. 4 Schematic drawing of a commercially available membrane contactor module





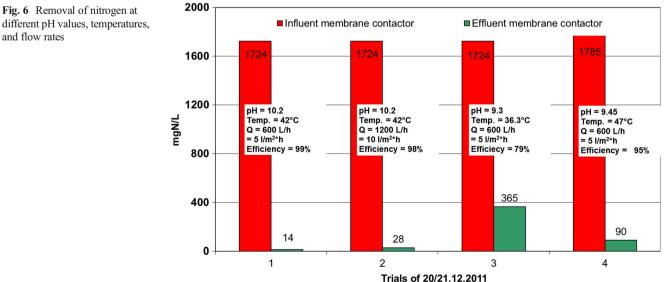
concentration differential across the membrane that acts as the driving force for removing ammonia from waste water. Figure 4 shows a schematic drawing of a membrane contactor module with a hydrophobic hollow fiber membrane bundle. The right picture shows schematically the chemo sorption process across a single hollow fiber. The wastewater flows through the shell-side of the membrane module (outside of the membrane), while the acid solution (sulfuric acid) is circulating on the lumen-side.

The central scope of the trials was to establish data on the elimination efficiency of the membrane, optimal temperature and pH, optimal flux (hydraulic load), long term behavior of the membrane, which pre-treatment of sludge water (filtration, CO₂-stripping, precipitation, etc.) are necessary, optimal flow scheme, and process configurations (multiple modules in series, multiple dosage of base, etc.).

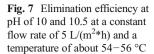
Results of the pilot scale tests with a membrane plant by Kunst GmbH (D) in 2011/12

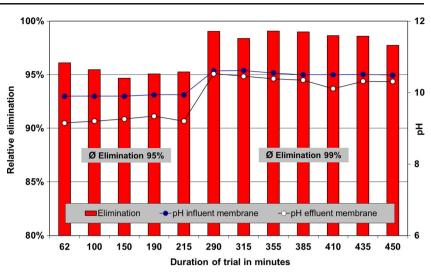
To get a first insight into the technology of free ammonia membrane stripping, in Winter 2011/12 single trials were conducted at WWTP Neugut (Switzerland) with a pilot membrane contactor by the German company Kunst GmbH (Heisele et al. 2014). The pilot plant consists of three membrane modules in series with a total surface area of 120 m^2 (40 m^2 per module). Two acid cycles were operated to evaluate the efficiency of two different membrane types (see flow scheme in Fig. 5).

In these tests, only the ammonium removal efficiency of the membranes itself were tested. To this end, three different sludge liquids with different qualities (nitrogen content in the range of 700 to 3,400 mg NH₄-N/L) of different WWTPs were treated with different hydraulic loads of 2.5 to $12L/(m^{2*}h)$:



and flow rates





- Supernatant WWTP Neugut: average N-concentration of about 1,700 mg N/L, P-concentration of about 300 to 450 mg P/L (activated sludge plant with enhanced biological phosphorous removal)
- Supernatant WWTP Altenrhein: average N-concentration of about 700 mg N/L
- Supernatant WWTP Batzenheid: average N-concentration of about 3,400 mg N/L (digester with co digestion of meat waste processing)

Figure 6 shows the removal of ammonium from the sludge water of WWTP Neugut at different pH values and

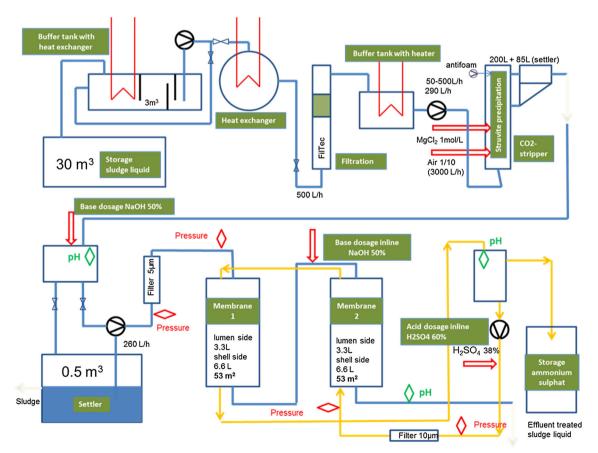
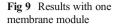
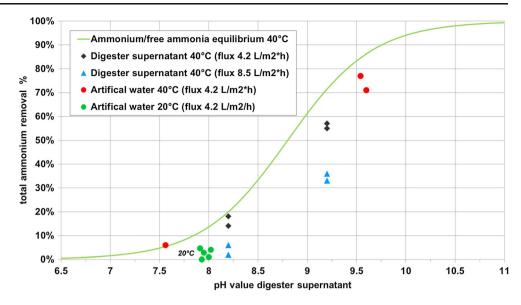


Fig. 8 Detailed flow scheme of the pilot scale plant of Sustec BV

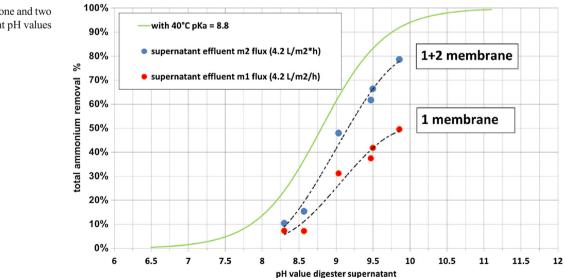


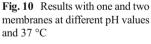


temperatures. In these trials, the removal of free ammonia was very successful. The elimination efficiency varied between about 80 to 99 %. At lower temperature and pH values, the elimination percentage was decreased. The trials were conducted at a flow rate of $600 \text{ L/(m^{2}*h)}$ (except trial 2 at 1.200 L/ $(m^{2*}h)$, corresponding to a specific flow rate of 5 L/ $(m^{2*}h)$ (trial 2 10 L/(m²*h)). A high elimination efficiency was observed in trial 1; nearly the same efficiency at high flow rates in trial 2. These results were due to the pH value of the waste water of 10.2. At these high pH values, nearly 100 % of the ammonium is deprotonated to free ammonia. The drawback is a high base consumption as a result of additional deprotonation of bicarbonate to carbonate. Due to both the lower influent temperature and the lower pH, the efficiency in trial 3 was lower (about 80 %). The higher temperature in trial 4 improved the efficiency again.

Figure 7 shows the eliminations efficiency of the membrane plant in a long term test (duration about 7.5 h). Also here, a high elimination was observed at high pH values (average influent concentration of 840 mg N/L, pH of 10 and 10.5, temperature in the range of 54 to 56 °C, flow rate 5 L/(m²*h)). The removal efficiency of the plant is nearly stable during the tests. Controlling the pH at 10.5 instead of 10 increased the elimination from about 95 to 99 %.

Trials with digester supernatant from WWTP Altenrhein showed similar results as with digester supernatant from WWTP Neugut. In these trials, a high flow rate of maximum 1,440 L/h (corresponding to a specific flow rate of 12 L/ $(m^{2}*h)$) was tested resulting in high ammonium elimination of 96% at a pH value of 10.4 and a temperature of 45 °C. Also at higher ammonium concentrations, the membrane





technology proved to be efficient. Trials with supernatant of WWTP Batzenheid with an ammonium concentration of about 3,400 mg N/L showed high removal efficiencies. With a high pH=10.4 to 11 and a low temperature (23.4 °C), elimination efficiencies between 84 to 97 % were reached.

Based on the test results, temperature and pH value are the dominant parameters for the removal efficiency of the membrane technology.

Results of the pilot scale tests with a membrane plant of Sustec BV (NL) in 2013/14

To get more experience with the membrane technology and to evaluate the long-term behavior (lifetime of the membrane, precipitation), optimal pre-treatment of the digester supernatant (filtration) and optimal operational parameters (temperature, pH, etc.) additional tests with a second pilot scale plant were conducted.

In autumn 2013, a half scale membrane contactor plant of the company Sustec BV was tested at WWTP Neugut. The membrane plant consists of two membrane modules in series (each module has about 51 m² surface area). In addition to ammonia separation by membranes, the recovery of phosphorous as magnesium ammonium phosphate before ammonia stripping was investigated. In this case, CO_2 stripping to increase pH and reduce base consumption is compounded with struvite precipitation. The flow scheme of the pilot plant is shown in Fig. 8.

In contrast to the pilot plant of Kunst GmbH, the plant of the Sustec BV offered the option to dose base in between the two membrane modules. Due to the removal of free ammonia in the first membrane module compartment, the pH value is decreased in the digester supernatant and can be raised again by this second dosage point.

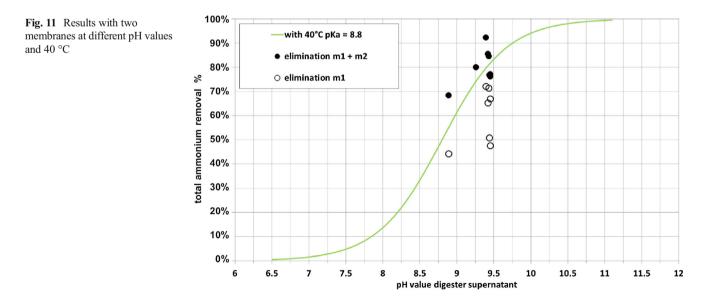
The first trials were done only with one module. In Fig. 9, the total ammonium removal of the membrane at different pH values and at a temperature of 20 and 40 °C is shown. The green curve shows the equilibrium ratio between ammonium and free ammonia at 40 °C in relation to the pH and indicates the maximum possible removal capacity of one membrane module, since only deprotonated ammonium can pass the membrane. These first results show that with a flow rate of 4.2 L/(m²*h), nearly 90 % of the free ammonia is removed. At flow rates of 8.5 L/(m²*h) (blue pyramid), the elimination efficiency is reduced.

Experiments with two membranes with no pH dosage in between the membranes (see Fig. 10) show that the elimination efficiency of the second membrane is reduced because after passing through the first membrane module, the pH of the sludge water is decreased. As a consequence, the elimination efficiency in the second membrane module is reduced due to a limitation of free ammonia.

Tests with two membrane modules in series and a dosage of base in between the modules demonstrated the same elimination efficiency in the two modules at the same inlet pH (see Fig. 11). After passage of the first membrane compartment, the pH was adjusted to the initial pH value of the inflow. The green curve shows the ammonium/free ammonia equilibrium at 40 $^{\circ}$ C and different pH values.

Outlook of the membrane technology

The results of the trials with two pilot scale plants showed a high potential of the gas permeable membrane technology.



Further tests in 2014 (especially long-term tests) will prove the reliability of the technology.

Acknowledgments This work was supported by the EU ROUTES project (Contract No 265156, FP7 2007-2013, THEME [ENV.2010.3.1.1-2] Innovative system solutions for municipal sludge treatment and management).

The authors would like to express their thanks to the operators of the wastewater treatment plants Kloten/Opfikon, Neugut, and Altenrhein for their support of our research.

References

- Boehler M. Liebi C (2012): Reduzierter NaOH-Verbrauch bei der Ammoniak-Strippung durch den Einsatz einer vorgeschalteten CO₂-Strippung – Betriebserfahrungen auf der ARA Kloten/ Opfikon, 8. Aachener Tagung zur Stickstoffrückbelastung, Heidelberg, 03.07. – 04.07.2012
- Boehler M, Schachtler M, Mohn J, Heisele A, Kunst W, Liebi C, Zullegg S, Siegrist H (2013): Full scale recovery of nitrogen from ammonia

rich sludge liquid and urine for the production of fertilizer by full scale air stripping or membrane stripping, extended Abstract, proceedings of 10th IWA Leading edge Conference on Water and wastewater technologies, Bordeaux, 2–6 June 2013

- Boehler M, Siegrist H, Seyfried A., Grömping M (2013): Ammonia Stripping from liquid side streams of sewage sludge processing and production of ammonium, book chapter 10 in Effective sewage sludge management-minimization, recycling of materials, enhanced stabilization, disposal after recovery, by Giuseppe Mininni, IRSA, CNR edizioni, Roma
- Heisele A, Boehler M, Siegrist H (2014) N-removal from sludge liquid by means of a gas selective membrane—half scale pilot trials at WWTP Neugut, summery report, in German
- Morales N, Boehler MA, Buettner S, Liebi C, Siegrist H (2013) Recovery of N and P from urine by Struvite precipitation followed by combined stripping with digester sludge liquid at full scale. Water 5(3): 1262–1278
- Udert KM, Larsen TA, Gujer W (2006) Fate of major compounds in source separated urine. Wat Sci Technol 54(11-12):413-420
- Ulbricht M, Schneider J, Stasiak M, Munoz J, Sengupta A, Kitteringham B (2009) Liquid-liquid extraction of ammonia using hollow fiber membrane contactors, Filtech conference, Wiesbaden, Germany, 13 – 15.10.2009