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# Operating problems in anaerobic digestion plants resulting from nitrogen in MSW

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## Abstract

Organic waste and municipal solid waste usually contain considerable amounts of different nitrogen compounds, which may inhibit anaerobic degradation processes and cause problems in the downstream and peripheral devices. This refers particularly to the different process stages of anaerobic digestion, to wastewater treatment, and to exhaust air treatment.

Neither the knowledge about nitrogen problems nor the technologies for elimination of nitrogen compounds from the wastewater or the exhaust air of anaerobic digestion can be regarded as state-of-the-art. Most of the technologies in question have already been applied in other areas, but are barely tested for application in anaerobic digestion plants. The few performance data and experiences at hand were mainly derived from pilot and demonstration facilities.

In this paper, the problem of nitrogen will be discussed in detail according to the separate problem fields based on the authors' experience, as well as on the basis of a review of the relevant literature. Furthermore, possible solutions will be proposed and the need for further research and development will be formulated.

## 1. Introduction

Due to the relatively low costs, the high flexibility of the process and the possibility of centralized and decentralized application, mechanical-biological waste treatment (MBT) processes are gaining importance, not only in Germany. In this context, anaerobic digestion for municipal solid waste treatment is becoming increasingly interesting due to its advantages in terms of energy production and exhaust emissions compared to aerobic procedures. Nevertheless, anaerobic digestion has not yet been able to establish itself on the market to the same extent as aerobic technologies. Apart from the higher investment costs for anaerobic digestion plants in comparison to aerobic treatment plants, this is also due to the fact that anaerobic digestion is still considered to be less stable in operation. Moreover, operational problems are more difficult to remedy, once they have occurred.

One important source of operational problems is the nitrogen compounds, which enter the process with the feed material. Table 1 shows the nitrogen content of different organic waste, sewage sludge and municipal solid waste. In the untreated raw waste, the nitrogen is predominantly organically bound.

Nitrogen may cause problems in anaerobic digestion because of its metabolic products:

- Ammonia ($NH_3$),
- Ammonium ($NH_4^+$),
- Dinitrous oxide ($N_2O$),
- Nitrite ($NO_2^-$),
- Nitrate ($NO_3^-$).

Fig. 1 shows the process areas and material stages in which nitrogen compounds may cause problems.

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Table 1
Physical–chemical parameters and nutrient contents of selected organic waste materials

| | $H_2O$ (% FM)[b] | Organic dry matter (% DM)[c] | $N_{total}$ (% DM)[c] | $P_2O_5$ (% DM)[c] | $K_2O$ (% DM)[c] | CaO (% DM)[c] | MgO (% DM)[c] |
|---|---|---|---|---|---|---|---|
| Organic waste | 52–80 | 34–81 | 0.6–2.1 | 0.3–1.5 | 0.6–2.1 | 2.2–6.8 | 0.2–1.7 |
| Green waste ("soft" organic) | 48–80 | 32–70 | 0.3–1.9 | 0.4–1.4 | 0.4–1.6 | 0.7–7.4 | 0.3–1.2 |
| Green waste (tree cuttings) | 25–52 | 65–85 | 0.1–0.4 | 0.1 | 0.3–0.5 | 0.5–1 | 0.1–0.15 |
| Sewage sludge (digested) | 65–85 | 15–40 | 4.0–5.3 | 4.7–5.2 | 0.3–0.5 | 5.7–8.2 | 0.8–1.2 |
| Bark | 45–75 | 60–85 | 0.2–0.6 | 0.1–0.2 | 0.3–1.5 | 0.4–1.3 | 0.1–0.2 |
| Kitchen waste | 75–95 | | | 1.0–1.5 | | | |
| Grape pomace | | 75–75 | 1.5–2.5 | 0.8–1.2 | 3.4–5.3 | 1.4–2.4 | 0.21 |
| Fruit pomace | 70–80 | 90–95 | 1.1 | 0.2– 0.6 | 1.57 | 1.1 | 0.2 |
| Rumen contents[a] | 10–20 | 80–90 | 1.3–1.2 | 1.1–1.6 | 0.5–0.6 | 2.0 | 0.6 |
| Paper | 25–30 | 62–79 | 0.2–0.8 | 0.15–0.6 | 0.02–0.1 | 0.5–1.5 | 0.1–0.4 |
| Draff/mash[a] | 90–95 | 90–95 | | 0.8–1.8 | | | |
| Yeast residues | 40–60 | 90–95 | | 1.4–2.0 | | | |
| Residual household waste (after separate collection) | 35–45 | 50–70 | 0.7–1.3 | 0.8–1.4 | | | |

[a] Kuhn (1995) and Weiland (1999).
[b] FM, fresh matter.
[c] DM, dry matter.

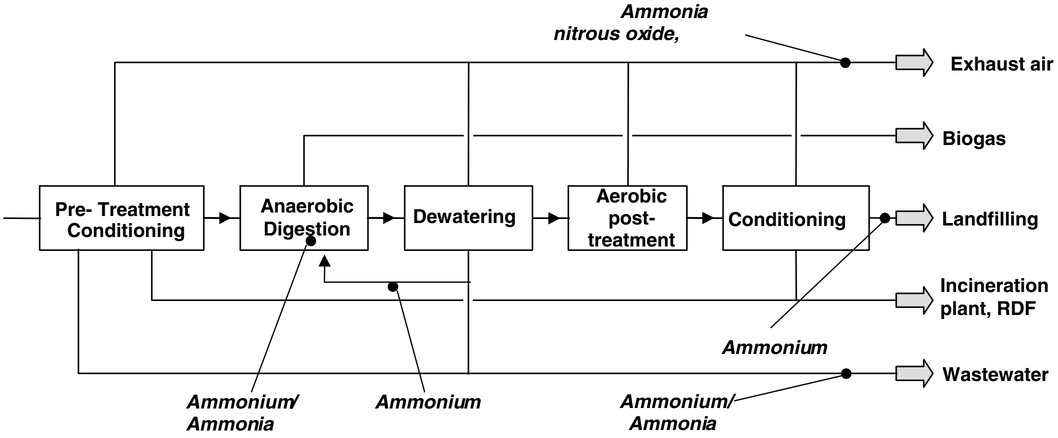

Fig. 1. Process areas and material stages of an anaerobic digestion plant possibly affected by nitrogen problems.

## 2. Biological process

The anaerobic digestion of organic matter is a complex process, which falls into four degradation steps. The specific microorganisms that take part in the process have different requirements on environmental conditions and moreover coexist in synergetic interactions. Nitrogen plays an important role in anaerobic digestion: Nitrogen is necessary for the formation of new biomass. Furthermore, in the form of ammonium, nitrogen contributes to the stabilisation of the pH value in the reactor. However, ammonium in high concentrations may lead to the inhibition of the biological process.

Microorganisms need nitrogen for the production of new cell mass, the absorption of nitrogen taking place in the form of ammonium. The nutrient requirement is low, which is due to the low biomass formation. A nutrient ratio of the elements C:N:P:S at 600:15:5:3 is sufficient for methanisation. As the reduced nitrogen compounds are not eliminated in the process, the C/N in the feed material plays a crucial role. The C/N should range from 20 to 30 in order to ensure sufficient nitrogen supply for cell production and the degradation of the carbon present in the process, and in order to avoid at the same time excess nitrogen, which could lead to toxic ammonium concentrations (Weiland, 2001).

Ammonium is an important parameter for the buffer capacity in an anaerobic reactor. With concentrations of up to 1000 mg/l, ammonium stabilises the pH value (ATV, 2002). Ammonium is released during the anaerobic hydrolysis of organic nitrogen compounds, causing an increase of the pH value. The ammonification thus counteracts the reduction of the pH value resulting from the acidification step of anaerobic digestion (ATV, 1993).

At a sufficiently high concentration, almost all substances inhibit anaerobic digestion (ATV, 1990). It should be noted that only the undissociated form of the intermediate catabolic product has an inhibiting effect on

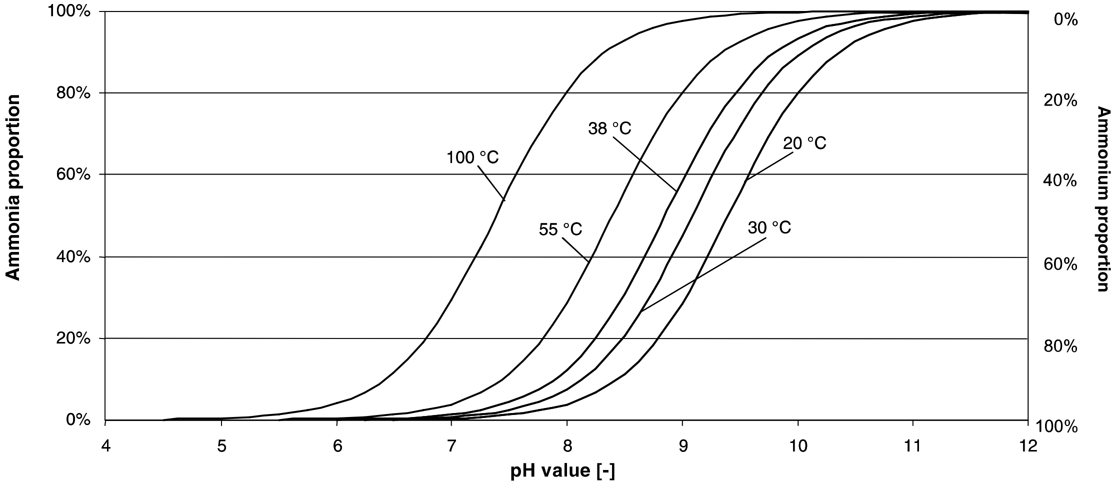

Fig. 2. Dissociation balance between ammonia/ammonium depending on pH and on temperature (calculated according to Kollbach et al., 1996).

microorganisms. As the dissociation equilibrium depends on the pH and on temperatures in the reactor, which both may vary, it is difficult to provide detailed data on toxic or inhibiting threshold concentrations. The dissociation balance of ammonia and ammonium, for instance, changes to ammonia with an increasing pH value and temperature as shown in Fig. 2. From this follows that even small changes in the pH value are sufficient to cause an inhibition. Furthermore, the bacteria may adapt themselves to high concentrations of certain substances, as long as the concentration of the respective substance increases slowly. Because of this situation, it is difficult to determine an exact threshold concentration that inhibits the process; rather broad ranges of possibly inhibiting concentrations can be given.

The ammonia-induced inhibition occurs primarily during the anaerobic digestion of organic waste materials, which are rich in proteins, as ammonia nitrogen is released through the mineralisation of organic nitrogen compounds. The range of inhibiting concentrations of ammonia is between 30 and 100 mg/l (at pH value $\leqslant 7$ and temperature $\leqslant 30$ °C), whereas the respective concentrations of ammonium are between 4000 and 6000 mg/l (ATV, 1990).

The inhibition effects by different intermediate catabolic products can counteract each other. With an increasing pH value, for instance, the inhibition by hydrosulphide and by volatile fatty acids declines, whereas the inhibition by ammonium nitrogen increases. With the presence of certain substances, the inhibition impact may even be reversed. In the presence of hydrosulphide and carbon dioxide, for instance, the dissociation balance of ammonia/ammonium is displaced in the direction of ammonium and the inhibition by ammonia is reversed (Knoche et al., 1996).

As mentioned above, microorganisms have the ability to adapt themselves to varying environmental conditions during a slow increase of, say, the ammonium or ammonia concentration in the reactor. Nevertheless, a sudden increase in the ammonia concentration leads to an inhibition of the biological process. There are different emergency measures for the rapid recovery of the process, as for example stopping the substrate supply, the addition of substrate with low nitrogen content, refeeding of digested material or lowering the pH value by addition of acids. All of these measures can only eliminate low degrees of inhibition. In the case of a high degree of inhibition, the only option is to empty the reactor and re-initiate the process. Therefore, close monitoring of the process is indispensable for early identification of inhibition effects.

In anaerobic digestion processes with intensive process water recirculation, ammonium may accumulate in the process water and thus in the substrate for anaerobic digestion with the effects described above. In that case, further measures for ammonium elimination may be necessary (see Section 4).

## 3. Exhaust air

The exhaust air emissions from waste treatment plants play a key role with regard to the acceptance by the population and the ecologic evaluation of the process. Nitrogen compounds that are relevant to the quality of the exhaust air are primarily ammonia and dinitrous oxide. In some countries, exhaust air emission quality and particularly emissions of odour and dinitrous oxide may be subject to permits.

In Germany, there are different legal licensing guidelines for the recovery of organic waste (“biowaste”), on the one hand, and for the treatment of municipal solid waste on the other:

- For the construction and operation of plants for *organic waste treatment* there are only essential requirements on exhaust air emission control, e.g., a minimum 300-m distance of these plants from populated areas and definition of maximum odour emissions of 500 odour units/m$^3$ (German Technical Instruction on Air Quality

Control -TA-Luft; Anonymous, 2002). These requirements can easily be fulfilled by structural measures and the treatment of the exhaust air from aerated windrows in a biofilter.

- In comparison to that, legal requirements on exhaust air emission control from *municipal solid waste treatment* are more detailed and stricter. The German 30th Federal Emissions Control Act (Anonymous, 2001) requires the complete encapsulation of the mechanical biological waste treatment plants, including exhaust air collection and treatment; it also defines limit values for some air pollutants, such as TOC, $N_2O$ and others (Table 2). In order to meet all of these requirements, a simple exhaust air purification in biofilters is not sufficient. As shown by research results and up-to-date operating experiences, it is necessary to treat the exhaust air in a thermal regenerative oxidation plant (TRO) in combination with an acid scrubber (Wallman et al., 2001).

The relevant exhaust air emissions of nitrogen compounds are ammonia and dinitrous oxide emissions. While ammonia contributes to the odour emissions and causes adverse effects on humans, the dinitrous oxide contributes to the anthropogenic greenhouse effect. Dinitrous oxide emissions can occur both during the intensive thermophilic phase of aerobic treatment/composting, and also during the aerobic post-treatment of solid digestion residues; this is equally relevant for the treatment of organic and residual waste.

Due to the mineralisation of organic nitrogen compounds under anaerobic conditions, the total nitrogen in the solid digestion residue is mainly present as ammonium and ammonia. In the first phase of aerobic post-treatment, most of it is stripped out in the form of highly volatile ammonia. The reduction of the ammonia concentrations in the waste by stripping with exhaust air leads to an increased release of ammonia from the ammonium fraction, as the proportion of ammonia and ammonium is in balance (see Fig. 2). In this way, up to 25% of the total nitrogen in the waste may be stripped out into the exhaust air.

The main ammonia emissions take place during the first week of post-treatment and can amount to up to 1000 mg/scm (standard cubic meters), as shown by the investigations of the aerobic post-treatment of digestion residues from a Valorga plant (IGW, 2001). In that investigation, the ammonia emissions declined in the course of further treatment and were about 100 mg/scm by the end of the third week. Similar evolutions of the ammonia concentration in the exhaust air were detected in other comparable investigations (Fricke et al., 2001).

Very high peak emissions of ammonia may occur if the temperatures in the windrows are high or if increased ammonium loads enter the aerobic post-treatment as a consequence of high ammonium contents in the solid digestion residue itself or in the process water used for the irrigation of the windrows. If the aeration is insufficient, high ammonia concentrations in the atmosphere of the treatment hall may result that exceed the German limit value for exposure at working places of 50 ppm. According to current operational experiences, only a strong, optimised suction aeration proved to be suitable in order to maintain the contamination of the atmosphere in the treatment hall below critical levels.

The ammonia in the exhaust air may be effectively removed through the use of an acid scrubber; the reduction rate can be close to 100%. Sulphur or nitric acids can be used as scrubbing acids, so that solutions of ammonium sulphate or ammonium nitrate are formed as products. So far, there is no established market for these products.

Table 2
Exhaust air emissions in comparison with the German limit values according to the 30th Federal Emissions Control Act (30. BImSchV – Anonymous, 2001)

| | German limit value (according to 30. BImSchV) | Exhaust air before treatment (aerobic treatment/ MSW composting) | Exhaust air before treatment (aerobic post-treatment of anaerobic digestion residues) |
|---|---|---|---|
| Exhaust air ($m^3$/ton waste input) (to treatment plant) | | 5000–9000 | 2000–6000 |
| TOC (mg/$m^3$)[a] | 20/40[b] | 50–200 (maximum up to 1000) | 50–200 (maximum up to 1000) |
| TOC (g/Mg) | 55 | 400–800 | 200–600 |
| Dinitrous oxide (g/ton waste input) | 100 | <100 | <100 |
| Odour (odour units/$m^3$)[a] | 500 | 10,000–30,000 (maximum up to 100,000) | 10,000–30,000 (maximum up to 100,000) |
| Polychlorinated dibenzo-*p*-dioxins (PCDD) and polychlorinated dibenzofurans (PCDF) (ng Toxicity Equivalents TE/$m^3$)[a] | 0.1 | $\ll 0.1$ | $\ll 0.1$ |
| Dust (mg/$m^3$) | 30/10[a] | $\ll 10$ | $\ll 10$ |
| Ammonia (mg/$m^3$)[a] | | 30–100 (maximum up to 500) | 100–300 (maximum up to 1000) |

[a] Referring to standard cubic meters (0 °C, 1013 bar).

[b] Daily/half-daily mean.

Nevertheless, the application as fertilizers in agriculture, given a sufficiently high nitrogen concentration, or the application for exhaust gas scrubbing during waste incineration are possible recycling options.

Thermal regenerative oxidation (TRO), which is applied for the oxidation of the organic carbons in the air, also leads to a reduction of ammonia. The reduction yield of ammonia is increased with higher temperatures in the combustion chamber of the incineration plant. Yet, as a consequence of ammonia incineration, the concentrations of nitrogen dioxides increase at the same time in the clean gas of the thermal plant (Wallman et al., 2001), leading to considerable odour emissions in the clean gas. In that case, the German limit value for odour emissions of 500 odour units/scm is usually exceeded. Acid ammonia scrubbing before thermal treatment of the exhaust air is thus indispensable in order to limit odour emissions to acceptable values.

As dinitrous oxide contributes to the anthropogenic greenhouse effect, special attention should be given to dinitrous oxide emissions of waste treatment plants. In Germany, the 30th Federal Emissions Control Act defines a limit value for dinitrous oxide emissions (Table 2). Depending on the source, a distinction can be made into the following types of dinitrous oxide emissions:

- Primary dinitrous oxide (in the exhaust air before treatment, formed during aerobic treatment) and
- Secondary dinitrous oxide (newly formed during thermal exhaust air treatment).

Primary dinitrous oxide may be formed in the aerobic post treatment of the solid digestion residues in the course of the microbiological degradation of organic matter.

It can thus be produced as an intermediate product of both nitrification and denitrification:

- Organic matter is decomposed by heterotrophic microorganisms, whereas the available concentration of ammonium increases with the degradation of the organic matter. This mineralisation of the organically bound nitrogen to ammonium takes place during anaerobic digestion and can be regarded to be a fairly completed process. The heterotrophic microorganisms in aerobic post-treatment need small amounts of ammonium nitrogen for the formation of new biomass, while the rest is freely available as ammonium nitrogen. A considerable amount of nitrogen is stripped off in the form of ammonia with the exhaust air. In the course of aerobic decomposition, the available carbon is reduced, thus the microbial activity of the heterotrophic microorganisms decreases as well. Consequently, the temperature in the windrows drops. The autotrophic nitrification bacteria grow preferably at lower temperatures, oxidizing or nitrifying the available ammonium nitrogen via nitrite and nitrate. However, oxidation of ammonium nitrogen to nitrate can be inhibited by high ammonia and nitrite concentrations. In particular, inhibition is aided by pH values over 7 and high ammonium concentrations in the feed material. Due to that, nitrite may accumulate, accompanied by the formation of dinitrous oxide (Wallmann et al., 2003).
- Dinitrous oxide can also be formed during denitrification, that is, during the reduction of the nitrate to elemental nitrogen. The formed dinitrous oxide is not inevitably emitted, because while passing through the windrow it can be further reduced to elemental nitrogen. However, this requires easily available carbon, which by the end of the aerobic treatment has almost completely been consumed.

In practice, it is very difficult to detect the origin of dinitrous oxide emissions if they occur. Typically, dinitrous oxide emissions are relatively low in the first two weeks of aerobic treatment. This is based on exhaust air measurements made by the authors at different aerobic treatment facilities of solid digestion residues in Germany (Fig. 3). High ammonia emissions at this stage of aerobic post-treatment may, however, lead to so-called secondary formation of dinitrous oxide in the thermal treatment of the exhaust air, as long as there is no acid scrubbing before thermal treatment. By the end of the second week of aerobic post-treatment, the concentrations of ammonia and dinitrous oxides in the exhaust gas show a reversed evolution: ammonia emissions decline while dinitrous oxide emissions increase.

In order to avoid these increasing dinitrous oxide emissions, the followings measures are recommended:

- During the first “intensive” phase of aerobic post-treatment, where the temperatures in the windrows increase to more than 60 °C, the digestion residues should be intensively aerated and turned as frequently as possible (aerobic stabilisation). The ammonium in the waste is thus stripped off as ammonia and can then be eliminated in an acid scrubber before thermal exhaust air treatment. This part of ammonium is no longer available for nitrification, so that less dinitrous oxide may be produced. Furthermore, the activity of the methanogenic microorganisms, which are present in the waste after anaerobic digestion, can be additionally reduced by intensive ventilation and regular turning.
- The frequency of turning can be reduced when the windrow temperature drops below 45 °C. However, when doing so the gas must be monitored for possible methane formation.

In conclusion it can be said that significant emissions of dinitrous oxide, which exceed the German limit value of 100 g $N_2O$/ton waste input, may occur if process control and monitoring are inadequate. With process control as described above, dinitrous oxide emissions should be kept far below critical levels as, for instance, defined by the German limit value.

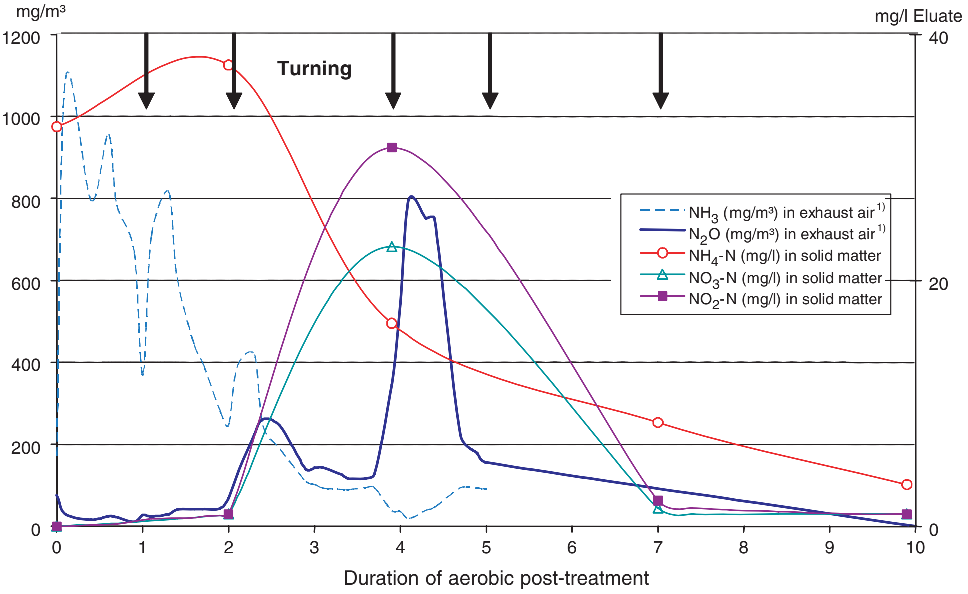

Fig. 3. Dinitrous oxide concentration in the exhaust gas (before treatment) and nitrogen compounds in the treated waste in the course of the aerobic post-treatment of solid digestion residues (Wallmann et al., 2003). [1] Referring to standard cubic meters (0 °C, 1013 bar).

## 4. Wastewater

In contrast to simple composting or aerobic treatment, relevant amounts of wastewater are generated during anaerobic digestion. It should be noted that the quantity of wastewater is basically independent of the type of process applied, but is determined by the moisture content of the waste input and the degree of dewatering after anaerobic digestion – unless no extra water is added to the process.

During anaerobic treatment of *organic waste*, approximately 200–500 l wastewater/ton waste input are generated (Loll, 1994; Gessler and Keller, 1995; Kübler, 1996).

In comparison to organic waste (“biowaste”), municipal *solid waste and residual waste* (after a separate biowaste collection) have as a general rule lower moisture contents, resulting in lower wastewater amounts of approximately 100–170 l/ton waste input (Fricke et al., 2004).

Tables 3 and 4 show selected analytical data of wastewater and process water from anaerobic digestion plants for municipal solid waste, residual waste and organic waste.

In many cases, wastewater generated during anaerobic digestion is recycled in the process. Nevertheless, the recycling of wastewater in the process is limited, on the one hand, by the quality of the recycled water. On the other hand, there will always be excess water as long as the water content of the waste output is higher than the water content of the waste input. Hence, all anaerobic processes have to integrate some solution for the treatment or disposal of wastewater.

- There are no specific requirements on the treatment of *wastewater from organic waste treatment* neither in the European nor in the German legislation. Up to now, no special attention has been drawn to the treatment of this wastewater, as most of it was recycled as fertilizer in agriculture or was sent to municipal wastewater treatment plants.
- Wastewater from municipal and residual waste treatment will thus usually require further treatment before disposal in water courses or in municipal wastewater treatment plants. Specific requirements might be defined in local legislation. In Germany, the Ordinance on the Treatment of Wastewater from Landfilling of Waste (*Rahmen-Abwasser-Verwaltungsvorschrift für die Reinigung von Abwasser aus der oberirdischen Ablagerung von Abfällen*) contains an annex on wastewater from MBT (Table 4). Most of the limit values defined there cannot be achieved without further treatment.

So far, only little consolidated experience with the design and operation of wastewater treatment in anaerobic digestion plants is at hand. Besides recycling in agriculture and further treatment in municipal wastewater treatment plants, different technologies known from sanitary engineering, such as ultra filtration, reverse osmosis, activated sludge and ammonia stripping, have been applied. In the context of a research project on the ecologically sound disposal of liquid manure funded by the German Federal Ministry of Education and Research, the applicability of these technologies for full or partial treatment of wastewater from organic and agricultural waste treatment was systematically studied (Hüttner and Weiland, 1997).

Below, the main outcome of the operational experiences and the research results with regard to nitrogen are presented, as well as more recent results from pilot and demonstration plants.

Table 3
Wastewater contamination from organic waste anaerobic digestion plants (Fricke et al., 2004)

| Unit parameter | Kautz and Nelles (1994) | Bidlingmaier (1995) | Kübler (1996) | Loll (1998) | Böning et al. (1999) | | Graja and Wilderer (1999); Widmann (1999) | Schmitt et al. (2001)[c] | Gallert et al. (2002)[d] |
|---|---|---|---|---|---|---|---|---|---|
| | [g/l] | [g/l] | [g/l] | [g/l] | [g/l][a] | [g/l][b] | [g/l] | [g/l] | [g/l] |
| Filtered solid matter | 4.8–15.7 | No data | 9.6–20.16 | No data | No data | No data | No data | No data | No data |
| $COD_{total}$ | 3.03–28.6 | 10.9 | 7.3–28.3 | 2.28–36.2 | 5.04 | 10.93 | 3.3–23.8 | No data | 4.46–5.80 |
| $COD_{dissolved}$ | 2.2–2.5 | No data | 2.18–4.90 | No data | No data | No data | No data | 45.2 | 4.10–5.25 |
| $BOD_5$ | 0.74–10.05 | 2.3 | 1.65–7.10 | 0.66–13.76 | 0.92 | 1.8 | 3.3–10.05 | No data | 1.88 |
| $N_{total}$ | No data | No data | No data | No data | No data | No data | 1.4 | 3.23 | 0.92 |
| $NH_4$–N | 0.23–2.0 | 0.61 | 0.51–2.6 | 0.57–1.49 | 1.18 | 1.74 | 0.23–0.98 | No data | 0.71 |

[a] Press water from dewatering of a mesophilic anaerobic digestion plant for bio-waste.
[b] Press water from dewatering of a thermophilic anaerobic digestion plant for bio-waste.
[c] Dewatering by means of a screw press.
[d] Dewatering by means of a decanter plus addition of flocculant.

As a consequence of anaerobic digestion, most of the total nitrogen in the output of fermentation is present as ammonium nitrogen and is organically bound only to a smaller extent. As the nitrogen content limits the recyclability, as well as the discharge to municipal wastewater treatment plants, nitrogen removal will usually be required. Table 5 shows the technical options of reducing the nitrogen content. Some of the processes have already been applied on a large scale for the treatment of industrial and municipal wastewater. The most relevant techniques and their applicability for the treatment of wastewater from anaerobic digestion are discussed in the following sections.

*Solid matter separation* is carried out for the dewatering of solid digestion residues in order to obtain solid material for composting or landfilling. Predominantly screw presses and decanters are used. In addition to that, belt presses can be applied for further solid matter separation if flocculants are added to the wastewater. Solid matter separation leads to a reduction of almost all relevant parameters and is particularly effective for contamination in insoluble, particulate form. The reduction of nitrogen compounds by solid matter separation is thus limited, because the most important nitrogen compounds, such as ammonium, are soluble. Solid matter separation yield differs considerably according to the applied aggregate. While screw presses with a screen have an elimination effect of between 15% and 20%, decanter centrifuges reach a separation effect of between 30% and 40% (Hüttner and Weiland, 1997; Rexilius, 1990).

The *membrane technology* was originally applied in the biochemical and pharmaceutical industries for the purification and recovery of valuable products and raw materials only, as the comparatively slow process requires sophisticated technology and high investment costs. The use of the membrane technology for wastewater treatment is a rather recent application.

According to the separated particle size, membrane technologies are divided into reverse osmosis, nano-filtration, ultra-filtration and micro-filtration. These differ additionally in the required membrane pressure differential (Fig. 4). Initially, mainly membranes on a cellulose basis were used. As the technology developed, they were substituted step-by-step by natural and synthetic polymers as a result of their better chemical and mechanical stability. Ceramic and metallic membrane materials, such as glass, play only a subordinate role.

In general, the membrane technology is suitable for the separation of both organic nitrogen compounds and ammonium nitrogen. However, nitrogen compounds are separated safely only in reverse osmosis plants. The processing of "raw" wastewater after solid matter separation in a reverse osmosis plant is not possible, as has been shown by several trial results (trials on wastewater of the biowaste treatment plants in Ottelfingen und Rümlang, Germany; trials on the treatment of wastewater from an agricultural biogas plant as part of a German federal

Table 4
Wastewater and process water contamination from anaerobic digestion stages of mechanical biological waste treatment plants

| Process | German Minimum requirements for waste water | | Böning and Doedens (2002) | | | | Schulze-Rettmer et al. (1992) | | Schmitt et al. (2001) |
|---|---|---|---|---|---|---|---|---|---|
| Unit parameter | Waste water for mixing (with other effluents), mg/l | Waste water for discharging in water courses, mg/l | Mesophilic | | Thermophilic | | Un-treated Liquid digestion residue, mg/l | Treated[a], mg/l | 1-stage thermophilic, mg/l |
| | | | Filtered, mg/l | Centrifuged, mg/l | Filtered, mg/l | Centrifuged, mg/l | | | |
| Solid matter | – | – | – | – | – | – | 2.4 | 1.6 | – |
| $COD_{total}$ | – | 200/400[b] | 3735 | 12,910 | 3763 | 15,534 | 6830 | 1023 | 99,350 |
| $COD_{dissolved}$ | – | – | – | – | – | – | 2290 | 780 | – |
| $BOD_{5\,total}$ | – | 20 | – | 1496 | – | 1978 | 2447 | 42 | – |
| $BOD_{5\,dissolved}$ | – | – | – | – | – | – | 427 | 14 | – |
| $NH_4$–N | – | – | – | 768 | – | 1036 | 1400 | 21 | 4153 |
| $NO_3$–N | – | – | – | – | – | – | – | 247 | – |
| $N_{total}$ | – | 70 | 860 | 1308 | 1214 | 1569 | – | – | – |
| $P_{total}$ | – | 3 | 16 | 60 | 17 | 62 | – | – | – |
| Mercury | 0.05 | – | – | – | – | – | – | – | – |
| Lead | 0.5 | – | 0.03 | 1.2 | 0.03 | 14 | 1.75 | 0.18 | 23.5 |
| Chrome/Chrome VI | 0.5/0.1 | – | 0.1 | 0.43 | 0.13 | 0.45 | 0.6 | 0.1 | – |
| Copper | 0.5 | – | 0.11 | 2.1 | 0.17 | 2.1 | 1.22 | 0.15 | – |
| Cadmium | 0.1 | – | – | – | – | – | – | – | 0.16 |
| Nickel | 1 | – | 0.18 | 0.36 | 0.22 | 0.41 | 0.73 | 0.33 | 3.1 |
| Zinc | 2 | – | 0.25 | 9 | 0.37 | 9.7 | 7.45 | 0.62 | 57.1 |
| Arsenic | 0.1 | – | – | – | – | – | – | – | – |
| Cyanide[c] | 0.2 | – | – | – | – | – | – | – | – |
| Sulfide | 1 | – | – | – | – | – | – | – | – |
| AOX | 0.5 | – | – | 0.59 | – | 1 | – | – | – |
| Hydrocarbons | – | 10 | – | – | | – | – | – | – |

[a] After nitrification/denitrification and further separation of solid matter.
[b] Limit value for “indirect discharging”.
[c] Highly volatile.

Table 5
Methods for nitrogen removal from wastewater of biowaste and municipal solid waste treatment plants

| Physical | Chemical | Biological |
|---|---|---|
| Solid matter separation | Precipitation | Nitrification/ denitrification |
| Membrane technology | Break point chlorination | |
| Adsorption | | |
| Desorption (stripping) | | |
| Evaporation | | |

research project on manure treatment and disposal). The fine dispersed particles still present in the wastewater cause rapid membrane fouling, clogging and mechanical damage to the membranes. Therefore, an additional up-stream membrane step has to be added in order to separate fine dispersed particles to a large extent.

Results of pilot wastewater treatment in an anaerobic digestion plant have shown that reverse osmosis alone only yields a nitrogen separation effect of about 95%, which is not sufficient in order to reach the strict German limit value of 70 mg/l total nitrogen (Table 6). The wastewater treatment plant implemented in this case included solid matter separation by means of a screw press and ultra-filtration. The insufficient reduction of nitrogen can be explained by the fact that the molecule form of the ammonia is similar to the one of water, which is why retention is insufficient. By adding acids, the ammonia can be transformed into ammonium, which shows a better retention rate. In this case, the nitrogen concentration in the discharge could be reduced to less than 10 mg/l. This corresponds to a separation yield of over 99%.

Wastewater treatment by the membrane technology shows the disadvantage that the produced concentrate has to be subjected either to disposal or to further treatment. A further volume reduction of the concentrate can be reached by evaporation, similar to the process applied for leachate treatment. In this case, treatment of the exhaust vapours is indispensable due to the enrichment with highly volatile organic compounds.

*Sorption* is the separation of gaseous components from a gas mixture by solvents. Desorption is the inverse process and results in the regeneration of the solvent. The *stripping of ammonia* from wastewater is a sorption process widely applied in wastewater technology. This method is preferably applied to the purification of industrial wastewaters

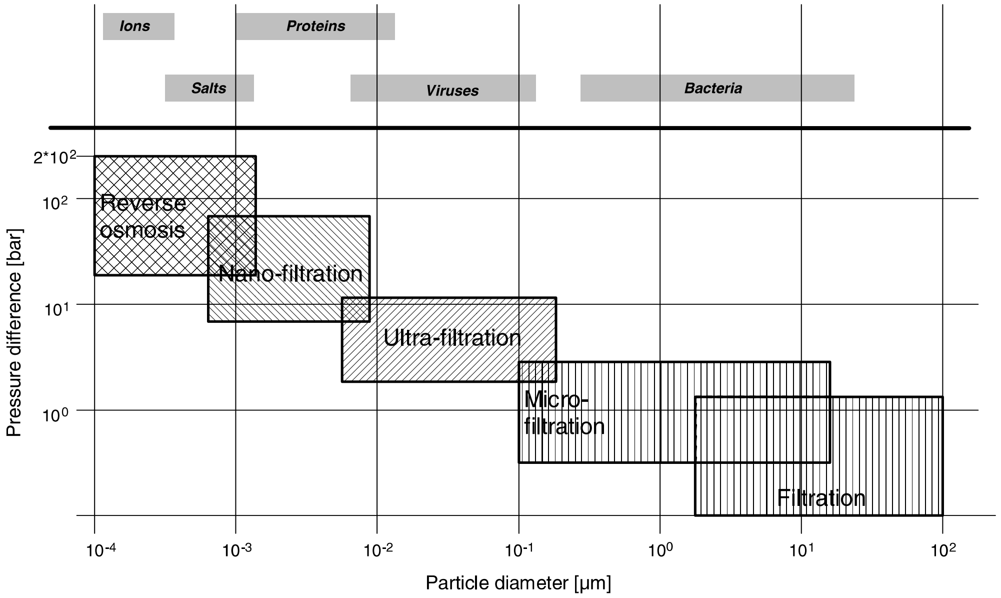

Fig. 4. Classification of membrane and filtration technology, as a function of pressure (modified according to Baumgarten et al., 1996).

Table 6
Average content of the permeate in a two-stage reverse osmosis for the treatment of the liquid digestion residue of an agricultural biogas plant (Hüttner and Weiland, 1997)

| Parameter | Influent [g/kg] | Effluent/permeate of reverse osmosis | |
|---|---|---|---|
| | | Without addition of acid [mg/l] | With addition of acid [mg/l] |
| COD | – | 32 | <15 |
| $N_{total}$ | 4.0 | 190 | <10 |
| $NH_x$–N | 2.9 | 156 | <10 |
| $NO_3$–N | – | Not detectable | Not detectable |
| $P_{total}$ | 0.6 | <2 | <1 |

contaminated with ammonia, such as from disposal of animal carcasses, or from municipal wastewaters, e.g., from sewage sludge digestion (Stein et al., 1995; Breitenbücher, 1996). The application for the reduction of ammonia concentrations in agricultural wastewaters has been researched in pilot plants (Hüttner and Weiland, 1997).

The reduction of nitrogen during stripping is limited to ammonia, whereas organic nitrogen cannot be stripped. Air or vapour is used as the stripping medium. In the process flow chart, stripping units are positioned after anaerobic digestion and solid matter separation, as solid matter can lead to technical problems in the stripping unit due to fouling and clogging. Displacement in the dissociation balance between ammonia/ammonium is achieved by the addition of alkaline chemicals in the case of air stripping, while during vapour stripping, the temperature is raised to 100 °C (see Fig. 6).

Vapour or air is supplied in counter flow to the wastewater, where it is charged with ammonia that will then be absorbed in an acid washing fluid. As a general rule, the stripping air is recycled in order to minimise the odour impact in the plant surroundings. Sulphuric acid is mostly used as a washing fluid. In this case, an ammonium sulphate solution is produced in the stripping unit. In vapour stripping plants with multiplier section, ammonium solutions with up to 25% ammonia can be produced. The ammonia in the wet solution can be further processed in a crystallisation stage to solid ammonium hydrogen carbonate.

Due to the requirements on media temperature and pressure, the investment costs for vapour stripping units are higher than the costs for air stripping units. As a general rule, the operational costs of vapour stripping units are determined to a great extent by the energy costs for

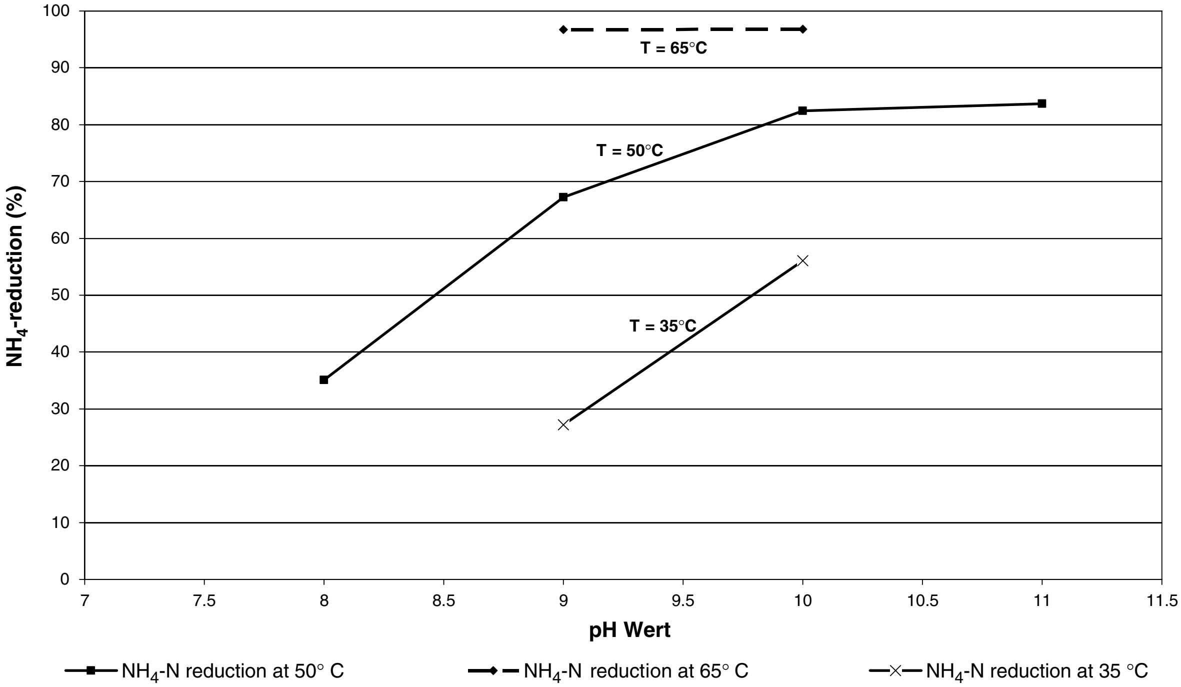

Fig. 5. Ammonium reduction by stripping at different temperatures as a function of pH (author's data, unpublished).

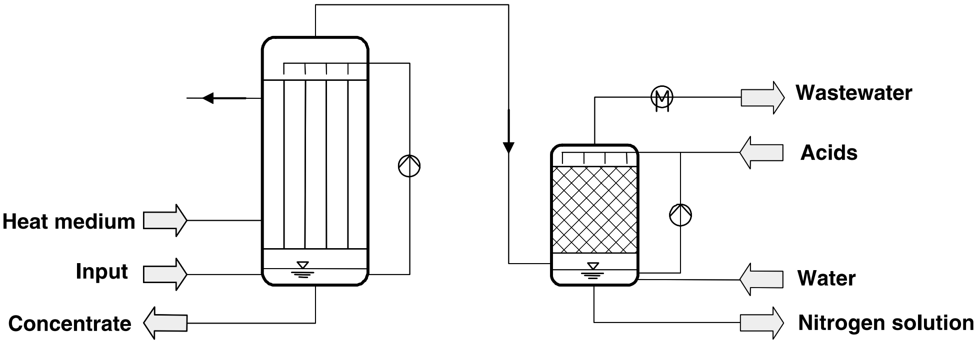

Fig. 6. Flow scheme for wastewater treatment consisting of an evaporator and an acid scrubber for the exhaust vapours.

vapour production. These costs can be significantly lowered if reasonable energy recovery concepts are applied or if free waste heat is available (e.g., from a CHP). Exhaust vapour compression is one of the methods widely used in practice for energy recovery.

In most cases, the pH value is increased by the addition of lime. The amount of chemicals required to increase the pH is strongly influenced by the composition and the buffer capacity of the wastewater to be treated. Trials with liquid manure with a high buffer capacity show that it is necessary to add about 30 kg sodium hydroxide per $m^3$ liquid manure for increasing the pH value to 11 (Schulze-Rettmer et al., 1990).

The achievable ammonium reduction in the wastewater depends on the temperature and the pH value, as presented in Fig. 5 for ammonia reduction by air stripping in liquid digestion residue after ultra-filtration. The results have been obtained during batch trails on a laboratory scale (batch volume approx. 7 l) at a constant aeration rate of 12.9 $cm^3$/min (standard cubic centimetres) and a retention time of approx. 3 h.

In summing up it can be said that selection of the stripping method, as well as determination of optimum operating parameters, has to take place as a function of:

- the wastewater that has to be treated (its quantity, ammonium concentration, pH value and buffer capacity),
- the required reduction effect, or the maximum permissible ammonium concentration in the output, and
- operational aspects, such as space requirements, heat and chemicals requirements.

The results of trials from air and vapour stripping plants are compared in Table 7. The concept of the air stripping plant was targeted at the total reduction of the ammonium, while in the vapour stripping plant only partial reduction was intended. The reduction yield of the vapour stripping plant can be considerably improved by increasing the amount of the vapour, so that a reduction rate of 99% can be reached, similar to air stripping.

Table 7
Results of air and vapour stripping trials (Weiland and Harmssen, 1993; Hüttner and Weiland, 1997)

| | Air stripping desorption column | Vapour stripping |
|---|---|---|
| $NH_4$–$N_{Input}$ (mg/l) | 3800–4800 | 2200–3000 |
| Through-put | | |
| Wastewater ($m^3$/h) | 1 | 3.6 |
| Stripping medium ($m^3$ air/h; kg vapour/h)[a] | 3200 | max. 450 |
| Temperature (°C) | 30–33 | 100 |
| pH (–) | 11.5–12.0 | 7.5–8.0 |
| $NH_4$–$N_{Output}$ (mg/l) | 10–30 | 300–800 |
| Reduction yield (%) | >99 | >80 |
| | | Absorption column |
| Fluid flow rate ($H_2SO_4$) ($m^3$/h)[a] | | 5 |
| Air flow rate ($m^3$/h) | | 3200 |
| pH (–) | | 2 |
| $(NH_4)_2SO_{4,Output}$ (%) | | 33–35 |

The processes of *precipitation and flocculation* often take place simultaneously, although these are two different chemical–physical processes. Precipitation means the transformation of dissolved, often ionic water compounds into an undissolved particulate form. Flocculation consists of the transformation of fine dispersed insoluble compounds to larger compounds suitable for mechanical separation by means of flocculation additives.

Ammonium nitrogen can be precipitated as magnesium ammonium phosphate (MAP) according to the following chemical equation:

$$NH_4^+ + Mg^{2+} + PO_4^{3-} \rightarrow MgNH_4PO_4$$

For this reaction, the other reactants magnesium and phosphate must be available in sufficient quantities according to the respective stoichiometric ratio. By the addition of magnesium oxide, this precipitation reaction can be initiated. The optimum pH value for precipitation is between 8.5 and 9.5. The purity of the produced MAP depends to a great extent on the precipitation conditions. Due to the heterogeneous composition of the wastewaters from anaerobic digestion, the MAP produced there will usually be contaminated with other salts.

Up to now, the precipitation of ammonium as MAP has not established itself in wastewater treatment. Apart from the high technical expenditures, this is mainly due to the higher costs of the precipitation process in comparison to biological ammonium reduction, at least if a high removal yield is required, as for example resulting from the German limit value of 30 mg/l. In that case, the revenues from selling the MAP cannot even cover the costs for the used chemicals (Schulze-Rettmer et al., 1992).

The precipitation of MAP may also occur spontaneously in the wastewater of anaerobic digestion plants, as generally all reactants are available in the wastewater. This MAP precipitation can lead to clogging and blocking. Although it is difficult to localise the endangered parts of the plant, experience shows that especially areas with high turbulences, such as for example the pipelines, the pumps or the degasification surfaces, are frequently affected.

In the treatment of wastewater by *technical evaporation*, the high volatility of the ammonia is used. In technical evaporation units, the volume of the aqueous solution is reduced by evaporation; the contaminants remain in the concentrate. As a general rule, the distillate shows low amounts of dissolved matter and low COD (chemical oxygen demand) values, the COD being mainly caused by high volatile organic components. Evaporation units are usually characterised by a high thermal energy demand. Furthermore, technical problems in the operation of evaporation units may occur due to the formation of films and fouling of the heat transfer units. These films are caused by caking of organic compounds, which are sensitive to temperature, or by the crystallisation of dissolved matter on the walls of the evaporation unit. As a consequence of fouling, the evaporation efficiency is decreased.

Depending on the configuration of the evaporation unit, the ammonium can be left in the concentrate or can be stripped with the vapour. The latter happens with molecular ammonia, which is then, after stripping, left in the condensate. Afterwards, the ammonia can also be stripped from the vapour as ammonium sulphate or ammonium nitrate with the aid of sulphuric or nitric acid in an additional acid scrubber. If acid is added to the wastewater input into the evaporation unit, ammonia will be transformed to ammonium, which is then – during evaporation – left in the concentrate (Hüttner et al., 1996).

The evaporation of wastewater from agricultural biogas plants has been analysed in connection with various demonstration-scale research projects. Vacuum falling film evaporators and vacuum horizontal spraying film evaporators were used, as these are highly suitable for the use of excess thermal energy that frequently is available at biogas plants. In the case studied here, excess heat with a temperature of about 70 °C was used. In one plant, ammonium retention was achieved by lowering the pH in the wastewater input; during evaporation, the ammonium was thus left in the concentrate. In the other plant, molecular ammonia was stripped with the vapour and then eliminated in an acid scrubber. With both plant configurations, the achieved ammonium retention effects were about 90%. Further increases in the retention yield seem to be possible, but were not required in the studied case. Furthermore, these trials show that with both plant configurations organic nitrogen compounds remain completely in the concentrate (Table 8).

The biological elimination of ammonium takes places by *nitrification/denitrification* of the nitrogen. The degradation of ammonium nitrogen to elemental nitrogen occurs by the intermediary step of nitrification according to the following chemical equation:

$$\mathrm{NH_4^+ + 2O_2 \rightarrow NO_3^- + 2H_2O + 2H^+}$$

with the aid of Nitrosomonas and Nitrobacter, which are ammonium oxidising micro-organisms. Here, nitrate is formed via nitrite formation. The latter, however, takes place so rapidly that usually only low residual concentrations of nitrite are measured. In order to prevent the process from being inhibited due to oxygen limitation, an operational oxygen content of more than 2 mg $O_2$/l is required. Furthermore, the produced hydrogen ions must be buffered.

Table 8
Material characteristics of the input and the exhaust vapour condensate of a plant for evaporation of the wastewater from an agricultural biogas plant (KTBL, 1999)

| Parameter | Input evaporator | Exhaust vapour condensate |
|---|---|---|
| COD (mg/l) | No data | 3100 |
| $N_{total}$ (mg/l) | 5700 | 40 |
| $NH_x$–N (mg/l) | 3800 | 40 |
| pH-value | 6.8 | 3.3 |

Table 9
Results from biological treatment of the wastewater of an organic waste treatment plant (Kübler, 1996)

| Unit parameter | Untreated [mg/l] | After flocculation and sedimentation [mg/l] | After biological treatment [mg/l] |
|---|---|---|---|
| Filtered solid matter | 5270–20,610 | 1390–2010 | 50–100 |
| $COD_{total}$ | 10,000–28,300 | 3000–5520 | 900–1380 |
| $COD_{soluble}$ | 2840–3830 | 1850–2610 | 820–1190 |
| $BOD_5$ | 4290 | 1450 | 80 |
| $NH_x$–N | – | 437–1142 | 1–35 |

The degradation of nitrate to elemental nitrogen takes place during denitrification under anoxic conditions, according to the chemical equation:

$$\mathrm{NO_3^- + 1/2H_2O \rightarrow 1/2N_2 + 5/2O + OH^-}$$

In order to achieve a high elimination effect, the environmental conditions have to be adjusted according to the needs of the involved microorganisms. In addition to an adequate oxygen content for the nitrification step, this comprises a pH value in between 7 and 8, as well as the availability of an easily accessible carbon source for the denitrification step. Furthermore, a stable biocenosis has to be formed, which is adapted to the specific nitrogen concentration.

Up to now, biological nitrogen removal from wastewater of solid waste treatment plants has mainly been studied in experimental and pilot plants (Table 9). In that context, the ammonium reduction yield is limited, as easily degradable carbon sources are normally lacking in the wastewater after anaerobic digestion. Thus, denitrification is often inhibited as long as no additional carbon source is added (Kübler, 1996).

This statement is also confirmed by more recent research results on nitrification/denitrification for treatment of the excess water resulting from anaerobic digestion of organically contaminated process water (Table 4) (Santen et al., 2003). The results show that the ammonium concentration in the process water can be reduced to up to 99% through biological treatment. However, in the treated wastewater, residual nitrate concentrations of 250 mg/l were detected, indicating insufficient denitrification. This inhibition of denitrification may be caused by inadequate process conditions as, for instance, aerobic conditions in the anoxic zones for denitrification. Nevertheless, this is more probably a consequence of the lacking carbon source in the wastewater, which has already undergone anaerobic digestion.

## 5. Summary

In the present paper, the operational problems caused by nitrogen compounds in anaerobic digestion and in the peripheral technology are explained. Different nitrogen compounds, such as ammonium, dinitrous oxide as well as nitrite and nitrate, can inhibit the biological process or make it difficult to comply with the corresponding emission limit values.

In the exhaust air, in particular ammonia, dinitrous oxide and odour play an important role. While ammonia contributes to the odour emissions and has adverse effects on humans, dinitrous oxide plays an important role in the anthropogenic greenhouse effect. Thus, these components should be reduced in the exhaust air. Experience with the treatment of exhaust air from aerobic post-treatment of solid anaerobic digestion residues shows that, with an acid scrubber and subsequent regenerative thermal oxidation (RTO), even the strict German limit values on exhaust air contamination can be met. In the case of biowaste treatment, fairly simple exhaust air treatment comprising an acid scrubber for ammonia elimination and a subsequent bio-filter for the reduction of odour, can be regarded to be sufficient.

Unlike “classical” aerobic biological waste treatment, relevant amounts of process and wastewater are generated during anaerobic digestion. As internal recycling of the process water is limited due to the contamination with various organic and inorganic compounds, and as there will always be some excess water, all anaerobic processes have to integrate some solution for the treatment or disposal of wastewater. In particular, nitrogen compounds limit the options for the recycling or discharge of the process water, so that as a general rule nitrogen elimination from the process water is required.

However, up to now the methods for nitrogen removal known from industrial and communal wastewater treatment are rarely applied in anaerobic digestion plants. Until now, the excess water from organic waste treatment plants has been used predominantly in agriculture or has been discharged to communal wastewater treatment plants. Neither is there any experience available with the treatment of process water from municipal solid waste treatment plants. Nevertheless, results from different research work and pilot scale applications show that established treatment technologies, such as reverse osmosis, evaporation and ammonia stripping, can also be applied for the treatment of process and excess waters from anaerobic digestion. In comparison to that, biological nitrogen elimination by nitrification/denitrification is in this case not that suitable, as denitrification is often inhibited by the lack of an easily available carbon source.

All in all, the material characteristics of the process and excess waters from anaerobic digestion differ considerably from the ones of communal and industrial wastewaters. Therefore, a simple one-to-one transfer of technologies, design and operation knowledge from communal and industrial wastewater treatment is as a rule not enough. In fact, the results and the efficiency rate of the selected technology must be tested in individual cases.

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