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Ammonia separation in novel bio filters

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Currently used bio filters are suited for odor reduction in livestock keeping but not for ammonia separation. Therefore it was the objective to develop a novel bio filter system which was able to ensure a high and long-lasting ammonia separation. This novel bio filter, which is equipped with a pH control in a water swamp beneath the filter layer and a conductivity control for water discharge, was investigated in terms of ammonia separation and nitrogen disposition over several months under practical conditions. The results show a stable ammonia separation of more than 88 % if certain consecutively described operating conditions are kept.

Keywords
Ammonia, bio filter, nitrogen balance, ammonia separation, pig keeping

Single stage bio filters are successfully used for decades to abate odor, inorganic and organic compounds in several economic sectors. For open bio filters maximum surface loads of 150 m³/(m² h) and maximum filter volume loads of 100 m³/(m³ h) are recommended with filling levels between 1.5 and 2.5 m (VDI 2016). In animal husbandry open bio filters are also used for odor abatement (Figure 1).

Figure 1: Single stage bio filters for odor reduction from animal houses (© J. Hahne)

Predominantly wood chips and fissured root wood have been proved of value. Maximum bio filter surface loads in livestock with 100 and 900 m³/(m² h) (TÜV 2009, Hartung et al. 1997, Hahne and Brandes 2002, Melse et al. 2014, Melse et al. 2015) and filling levels from 0.25 (wood chips) to 1.4 m (fissured root wood) clearly exceed those of industrial applications in many cases. A reliable separa-

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tion of odor and particulate matter has been demonstrated in a long-time test for a maximum filter load of 440 $m^3/(m^2\ h)$ and a 12 month old filter material (wood chips) (DLG 2007). In pig keeping the named maximum surface load do only appear under summer conditions (outside temperature > 26 °C) and full-grown animals. In annual mean the surface load is about 45–50 % of the maximum load. Keeping the requirements of ventilation (DIN 2004) and indoor air quality (TierSchNutztV 2001) and depending on species and housing technique, the exhaust air from animal husbandries shows odor concentrations between 40 and 5,000 OU/m^3, total dust and ammonia concentrations between 0.1 and 17 mg/m^3 and 0 up to 30 mg/m^3, respectively. These comparatively low concentrations result from high air volumes in livestock which are required for removal of heat, humidity and trace gases. The concentrations of organic and easy degradable gaseous carbon compounds are very low with less than 3 mg/m^3 total carbon. Thus, the exhaust air the carbon-to-nitrogen ratio (C : N ratio) offers a considerable nitrogen surplus, because the C : N ratio in biomass is 10–20 : 1. In this respect the surplus nitrogen can't be fixed in biomass.

The possible ammonia separation in bio filters is assessed as being of limited suitability (VDI 2016). Several investigations on bio filters working under practical conditions in livestock husbandry show that this assessment is correct. Week-long measurements on different bio filter systems (open bio filters with wood chips, straw beds and root wood) in practice showed ammonia separations between 49 and 67 % (wood chips), −9 up to 23 % (straw beds) and 60 up to 82 % (root wood) (Trimborn 2006). The ammonia separation was accompanied with an increase of N_2O emissions amounting to 3–17 % of the separated ammonia. At a fattening pig unit an average ammonia reduction of 42 % (n = 6 measurements over the year) was determined with a bio filter filled with a layer thickness of 25 cm new wood chips. The mean filter surface load was 341 $m^3/(m^2\ h)$ (Melse et al. 2014). Comparable measurements at another bio filter with an average surface load of 276 $m^3/(m^2\ h)$ resulted in a mean ammonia reduction of 38 % (Melse et al. 2015). A TÜV Rheinland evaluation of measurements in pig keepings showed for specific bio filters ammonia separation values between 49 and 94 % with surface loads from 100 to 907 $m^3/(m^2\ h)$ (TÜV 2009). Elder long-term investigations on bio filters which were dimensioned for a maximum surface load of 340–380 $m^3/(m^2\ h)$ and equipped with a mixture of coconut fibers and fibrous peat showed in summary that the mean ammonia separation varied between −11 and +36 %. The separation efficiency declined considerably with increasing surface load (Hartung 1997). Own investigations on a test bio filter, which was equipped with a 0.53 m bark mulch layer and used to clean exhaust air from a pig stable, showed an ammonia reduction between 87 and 95 % over a period of 148 days. But at the same time considerable amounts of secondary trace gases (NO_x-N, N_2O-N) were produced. The filter surface load varied between 108 and 295 $m^3/(m^2\ h)$. A nitrogen balance showed with a recovery rate of 86.3 % that 31.6 % of the input nitrogen was released with the outlet, 26.2 % was accumulated in the filter material and 28.5 % was regained in the water reservoir (Hahne and Brandes 2002).

A considerable amount of ammonia arises in the litter-free conventional pig keepings, where many bio filters are used for odor reduction. With an ammonia emission factor of 3.64 kg per pig place and year (VDI 2011) a fattening pig stable with 1,000 heads produces 3,640 kg of ammonia per year. Supposing 70 % ammonia reduction efficiency, the bio filter might accumulate 2,548 kg ammonia (2,098 kg N) as ammonia, nitrite, nitrate and organic fixed nitrogen every year.

The Hagola bio filter, which was approved by the Deutsche Landwirtschaftsgesellschaft (DLG), can be operated with a maximum filter surface load of 440 $m^3/(m^2\ h)$ (DLG 2007). On basis of manufacturer's data a maximum volume flow of 88,000 m^3/h has to be calculated for 1,000 pigs and thus a filter area of 202 m^2 is required. The biologically working wood chips layer is 30 cm high resulting in a filter volume of 61 m^3 and 48 tons, respectively, if the filter material is saturated with moisture. Because the filter material will be mineralized during useful life and the water content can't be increased as well, the nitrogen content (N) in the filter material with a water content of 70 % might increase calculative by 146 g/kg dry matter. But this is not the case. Neither the nitrogen can be fixed endlessly in organic mass nor accumulated in the humidity layer of the filter material. The maximum found total nitrogen concentration (sum of all mineral nitrogen compounds + organic fixed nitrogen) in the wood chips (analyzed via mixed samples from six single samples distributed over the whole layer thickness and the filter area) was nearly 60 g/kg total solids (Hahne et al. 2016). Because the nitrogen content in the wood chips do demonstrably not rise in the same degree as required for a significant ammonia separation in pig keeping, it has to be assumed that nitrogen is released via ammonia, nitrous and nitric oxides in the outlet air. A heterotrophic denitrification resulting in molecular nitrogen can be assessed to be negligible because of high air volumes and a corresponding oxygen supply in the filter material and a low concentration of easy degradable carbon as well. Based on these considerations and the completed approval tests it becomes evident that conventional single stage bio filters do not ensure a permanent ammonia separation in livestock.

Therefore is was objective of the work to develop a bio filter system and testing it in the frame of a DLG test, which secures a permanent and reliable ammonia separation. In addition to that the nitrogen remaining should be clarified with nitrogen balances.

Reference unit, process description, methods and test procedure

The novel bio filter system (Figure 2) differs in following essential issues from conventional bio filters:

- Beneath the pressure chamber the system is equipped with a water level control in a water reservoir. It is refilled with fresh water and kept on a constant level.
- A regular and intermittent irrigation is carried out from the water reservoir. The irrigation intensity is adjusted that way that irrigation water regularly leaves the filter material and flows back to the water reservoir.
- The pH value of 6.5 in the water reservoir is controlled with sulfuric acid to avoid an ammonia breakthrough by too high pH values.
- An automatic water discharge takes place if the conductivity in the reservoir exceeds 25 mS/cm.
- The active bio filter material (wood chips) has to be changed every six month due to the nitrogen accumulation.

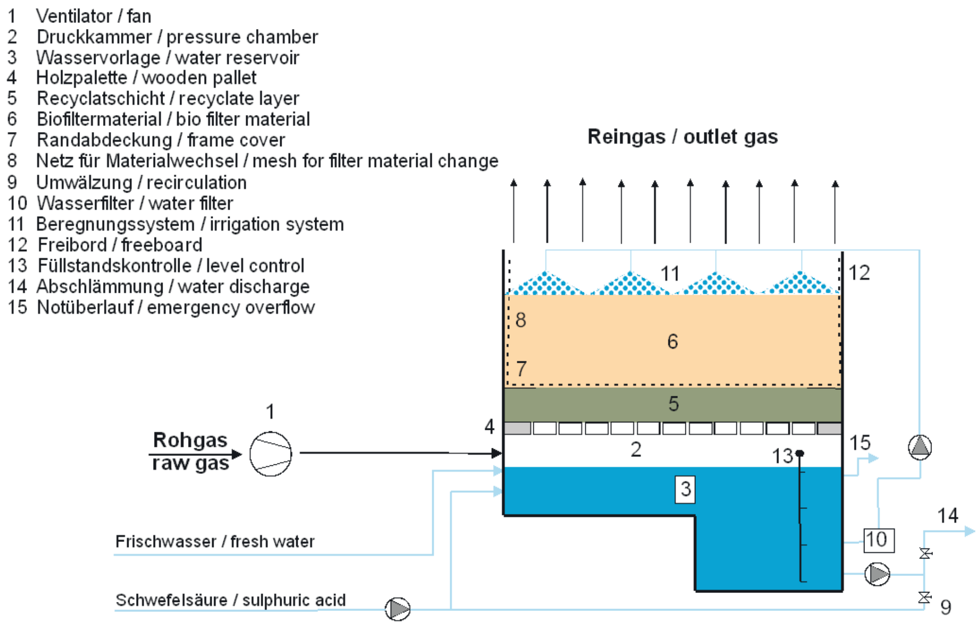

Figure 2: Process design of the new bio filter

The raw gas enters via the ventilation system (1) the pressure chamber (2) above the water reservoir (3) and flows initially through a wooden pallet (4). Coarse particles deposit via inversion of the air flow direction. Afterwards the raw gas flows through the filter material (6), consisting of a 6 cm plastic recyclate layer (5), which serves as a permanent growth area for microorganisms, and an above located layer of 30 cm wood chips. A frame cover (7) is installed to avoid wall effects. The wood chips material is placed above a mesh (8) to enable a rapid and automatic exchange of the filter material. The pH value in the water reservoir beneath the filter is adjusted to 6.5 by dosing sulfuric acid. A time-controlled recirculation secures the mixing of the water in the reservoir (9). The water is pumped through a water filter (10) into the irrigation system (11). A sufficient dimensioned freeboard (12) prevents the release of aerosols during bio filter irrigation. Ideally the water content of the wood chips should be between 60 and 70 %, which can be achieved with surplus irrigation. The filling level of the water reservoir is controlled via sensors (13). Evaporation losses will be balanced by an automatic fresh water dosing. If the conductivity of the irrigation water exceeds a maximum value of 25 mS/cm, a portion of it is discharged (14). An emergency overflow (15) secures a reliable exhaust air treatment, even at heavy rains.

The investigations were conducted at a forced ventilated, fully slatted floor pig stable with 312 fattening pig, which were kept in small groups. Liquid dispensers were used for feeding. The over-floor ventilation was implemented by means of two fans in a central air exhaust duct in the attic. The bio filter had a surface area of 70.84 m^2 and the maximum filter surface load was 440 $m^3/(m^2\ h)$. Additional specifications are summarized in Table 1. The measurements were carried out in the periods from 21 July 2015 to 22 September 2015 (summer measurements) and between 25 January 2016 and 22 March 2016 (winter measurements), respectively. The wood chips layer was exchanged on 28 September 2015 as scheduled.

Table 1: Specifications of the reference system for 312 fattening pigs

Parameter	Unit	Value
Filter surface area	m^2	70.84
Maximum filter surface load	$m^3/(m^2\ h)$	440
Maximum filter volume load	$m^3/(m^3\ h)$	1,486
Irrigation density	m^3/h	0.6
Water reservoir	m^3	14
Fresh water consumption	$m^3/(TP\ a)$[2)]	1.5[1)]
Water discharge	$m^3/(TP\ a)$	0.44[1)]

[1)] Calculated annual mean.
[2)] AP = Animal place.

The measurements methods according Table 2 were applied.

Tabelle 2: Messverfahren

Parameter	Method
Volumenstrom	2 measuring fans, Stienen (diameter 820 mm)
NH_3, N_2O	FTIR analysator, Gasmet Cx 4000[1)]
NO_x	DIN EN 14792 Chemilumineszenz Horiba PG 350E [1)]
NH_4-N	DIN 38406-E5-2
NO_2-N, NO_3-N	EN-ISO 10304-2
Total solids	DIN EN 12880
Organic N	DIN EN 25663
pH value	Hach-Lange HQ40d
EC	Hach-Lange HQ40d

[1)] The gas analyses were made by LUFA Nord-West.

The volume flow and ammonia concentrations in raw and outlet air were analyzed online over the whole period. The outlet volume flow was formally equalized with the inlet flow. Nitrous oxide and nitric oxides were measured online during the periods of nitrogen balancing. For a representative gas sampling the bio filter was encased. The point of gas sampling which was equipped with an insulated and heated pipe was located in the central area of the bio filter 1.5 m above the wood chips layer (Figure 3). The bio filter outlet was implemented over the whole face side of the bio filter which is not visible in Figure 3.

Figure 3: Roof construction for representative gas sampling

Generally the irrigation water sampling was carried out weekly after mixing the water reservoir. For nitrogen balancing samples from start and end of the measuring periods were taken. The filter material was sampled in intervals of 2–4 weeks, in which six single samples – two from the upper zone, two from the intermediate and two from the lower zone of the wood chips layer – were assembled to a mixed sample which was then mechanically hackled. For the analysis of nitrogen compounds the samples were eluted over 6 hours with distilled water. After centrifugation and membrane filtration pH value, electric conductivity, ammonium, nitrite and nitrate were measured in the eluate. The Kjeldahl nitrogen and the dry matter content analyses were carried out from the eluted solid matter.

Results

With a mean livestock of 308–312 animals the ammonia concentrations and the ammonia mass flows as well showed annual variations (Table 3). In summer the mean ammonia mass flow was 2.8 higher than in winter. Particularly this result can be led back on the volume flow which has been three times higher than in winter. The mean raw gas temperature was 22.8 °C in summer and 19.0 °C.

Table 3: Raw gas ammonia concentrations and mass flows

Periode	Parameter	Unit	Minimum	Maximum	Mean	Standard deviation
Summer	NH_3	ppm	7.3	25.0	12.8	3.1
n = 2,918	V	m³/h	12,639	38,377	30,459	6,714
	m	g/h	173.6	425.3	263.2	39.7
Winter	NH_3	ppm	8.1	22.2	13.4	2.2
n = 2,756	V	m³/h	4,652	2,0270	1,0167	2,625
	m	g/h	50.6	224.5	94.0	19.5

V = volume flow
m = mass flow

Under these circumstances and after an increase of the wood chips water content to 60 % the mean ammonia separation efficiency was at least 90.8 % during the summer measuring period (Figure 4 and Figure 5). At the measuring starting point the filter material was 6 weeks old. The full performance of the filter material was barely achieved at the starting point (Figure 4). Taking into consideration only the period beginning from early August (n = 2,405) the mean ammonia separation was 92 ± 3.5 % with minimum and maximum values between 80 and 100 %. The run-in period can be shortened surely if the exchange of the filter material would be done with pre-humidified wood chips.

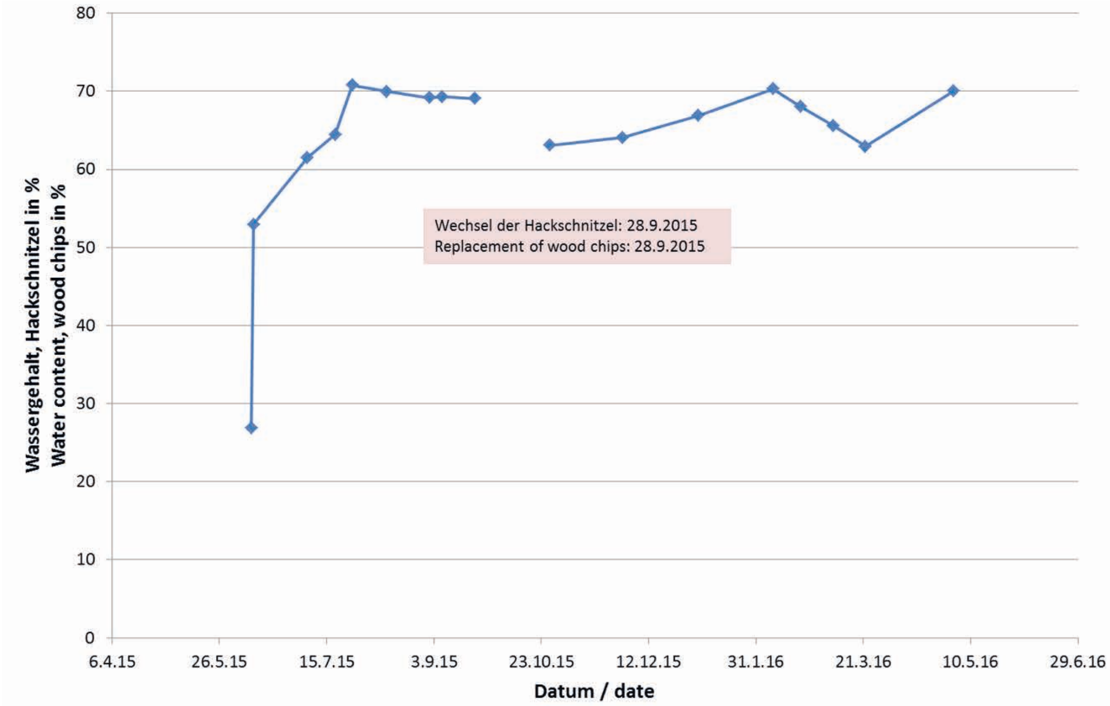

Figure 4: Temporal course of the water content in the wood chips layer

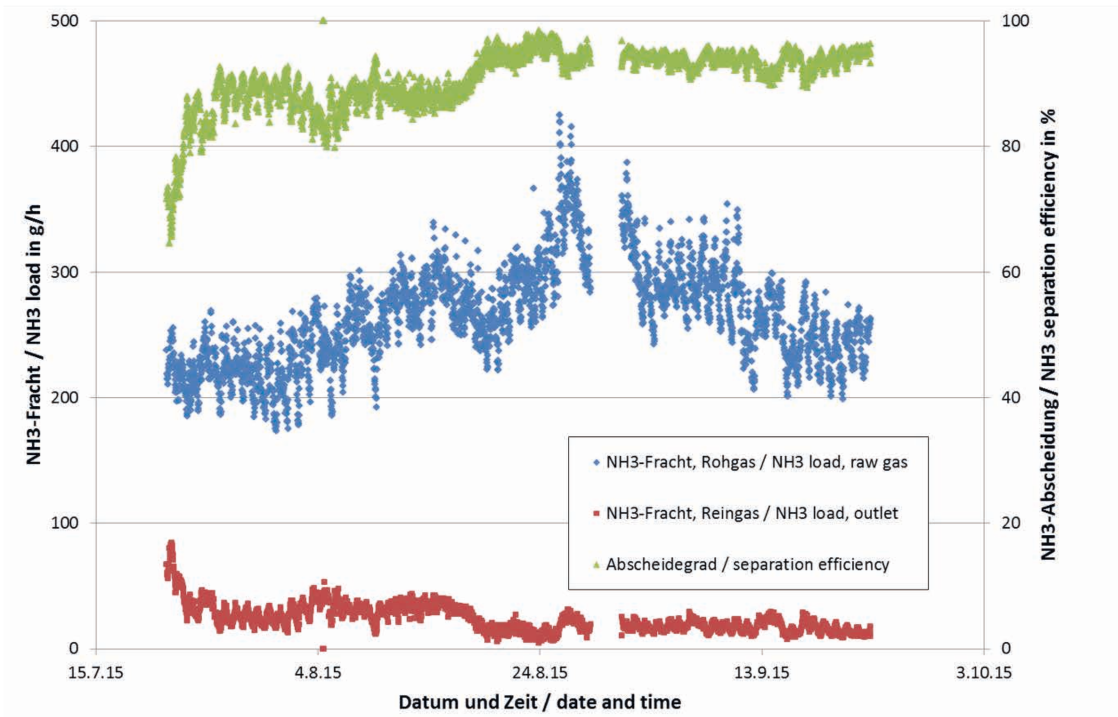

Figure 5: Raw and outlet ammonia mass flows and separation efficiency during summer measurements

After summer measurements the filter material was exchanged on 28 September 2015 and then the system operation was continued until starting the winter measurements. The winter measurements started on 25 January 2016 with a four month used filter material. During winter measurements (Figure 6) the minimum ammonia separation efficiency was 92.9 % and the maximum separation efficiency was 98.7 % under consideration of 2,756 pair of values (total measuring period). The mean separation efficiency was 97 +/- 0.9 %. Presumably the main reason for the improved ammonia separation during winter under comparable operation conditions is the threefold lower volume flow and the corresponding prolonged residence time.

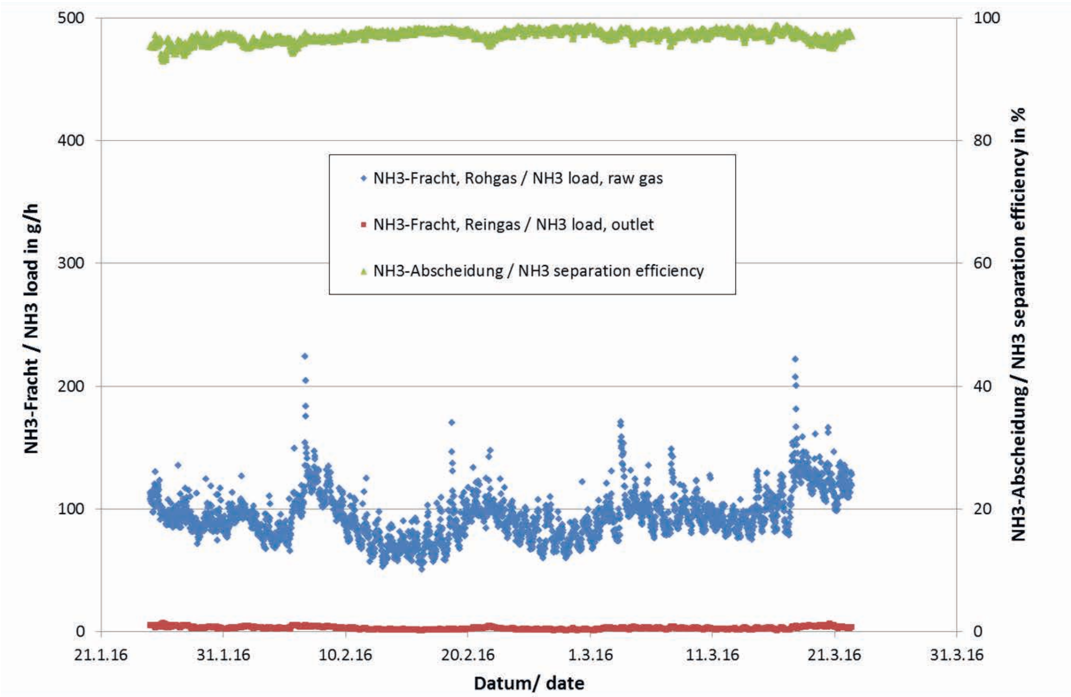

Figure 6: Raw and outlet ammonia mass flows and separation efficiency during winter measurements

The measurements in the frame of nitrogen balancing periods showed that the ammonia from the raw gas was accumulated in different forms in the filter material, in the water reservoir and in the discharge water (Table 4). About 20 % of the NH_3-N input was accumulated in the filter material and 60 % in the water reservoir and the discharge water. A heterotrophic denitrification can be excluded widely because of a lack of easy available organic carbon and the continuous aerobic conditions. During summer measurements not any secondary trace gases arose in the bio filter outlet. The mean ammonia separation efficiency was very good with 93 % at least. During the winter balancing period nearly 8 % of the NH_3-N input was detected as secondary trace gases. The nitrous concentration in the outlet and the nitric oxides concentrations as well increased about 0.3 ppm and 0.2 ppm each in average, compared to the inlet concentration. Figure 7 shows the courses of the different trace gas concentrations during the winter measurements. Concerning nitrous oxide and the nitric oxides the net concentrations (outlet minus inlet concentrations) were illustrated. In spite of a low secondary trace gas production the N-separation during winter was also very good with nearly 90 %.

Table 4: Results of nitrogen balances during summer and winter

Period	N input in kg	N accumulation BF in kg	N accumulation W in kg	N clean gas in kg	N separation in %
Summer[1)]	236	47	140	16	93
Winter[2)]	48	10	27	5	90

[1)] 11.08. to 22.09.2015.
[2)] 09.02. to 08.03.2016.
BF = bio filter
W = water

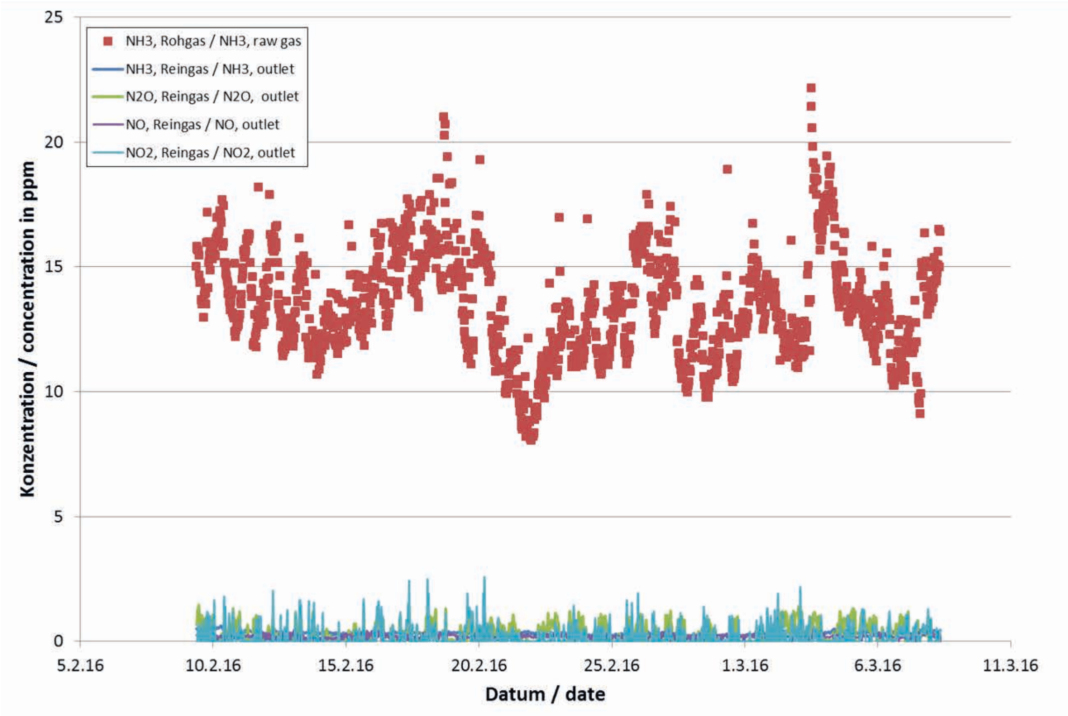

Figure 7: Temporal course of ammonia in raw and outlet gas and the net nitrous oxide, nitrogen monoxide and nitrogen dioxide concentrations in the outlet during the winter measurements

Besides the ammonia separation which is calculated by cumulating the ammonia mass flow in raw and outlet gas, the "real" nitrogen removal is essential in terms of environmental effects. The "real" nitrogen removal means the percentage ratio between the removable amount of nitrogen and the nitrogen mass which enters the bio filter with the raw gas. The discharge water and the nitrogen enriched bio filter material can be considered as removable nitrogen. During summer the nitrogen removal was 79 % and during winter it was 78 %.

In this proper dimensioned and moistened bio filter raw gas nitrogen will be separated and accumulated in the bio filter material in two ways via ammonium assimilation and ammonia oxidation as the measurements confirm (Figure 8). During the summer measurements the total nitrogen concentration in the wood chips layer increased fivefold within a period of 3.5 month and achieved a concentration of 18.5 g/kg total solids. After the summer measurements the wood chips layer was regularly exchanged because of this nitrogen accumulation. The winter measurements were carried out without an exchange of the filter material and resulted in lower nitrogen enrichment as compared to the summer results. This finding communicates well with the significant lower nitrogen mass flow during winter (Table 3). Nitrite arose only short-term in the measuring period. The NH_4-N : NO_x-N ratio varied between 1.3 and 2.9 and was 1.9 in mean of all measurements (n = 41).

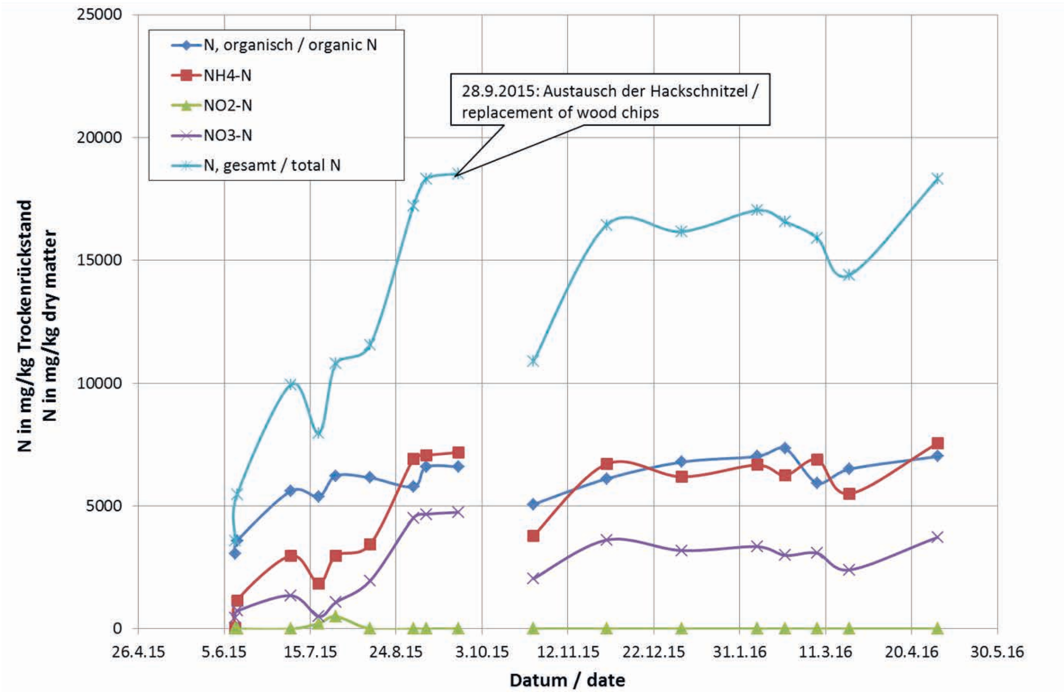

Figure 8: Accumulation of different nitrogen species in the wood chips layer

The wood chips layer is subject to an acidification which can be attributed to the oxidation of ammonia in particular. Low pH values and increasing nitrite concentrations in the filter material lead to increasing nitric oxides (NO_x) and nitrous oxide (N_2O) emissions as well. During the nitrogen balancing in winter about 8 % of the NH_3-N input was released as secondary trace gases while no equivalent increase could be detected during summer. This finding communicates well with the decreasing pH values in the wood chips layer during the winter measurements (Figure 9). The rapid decrease of the pH value in winter could be led back on the considerable lower NH_3-N input during winter (Table 4).

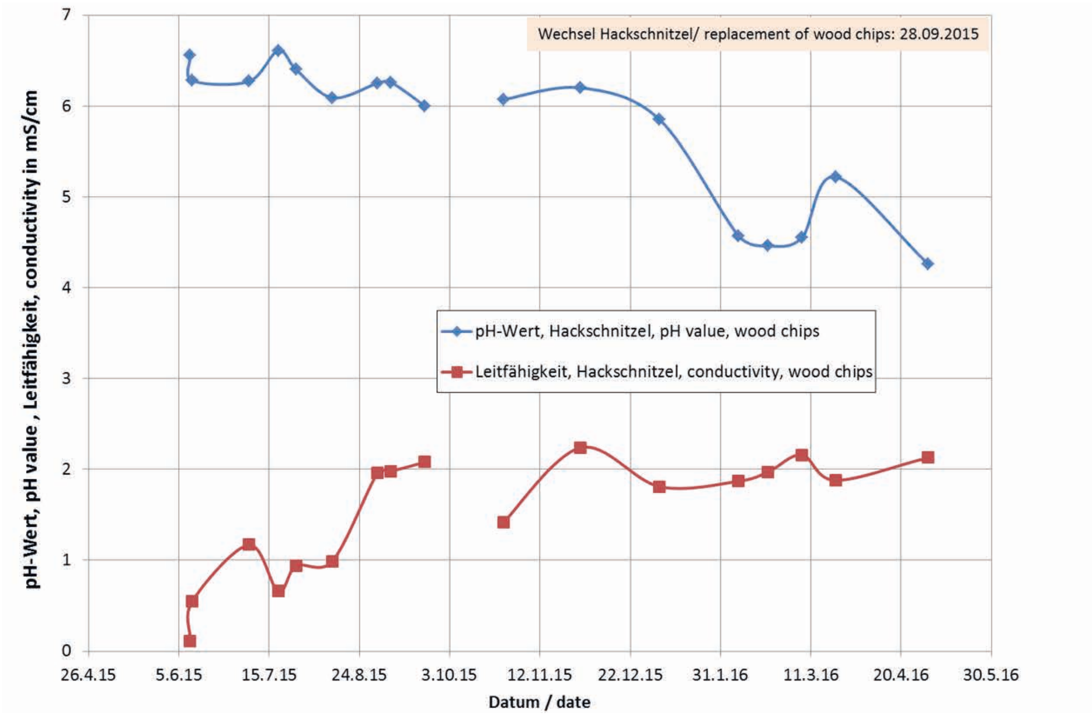

Figure 9: Course of pH value and electric conductivity in the wood chips layer

Nitrogen has to be removed regularly from the bio filter system to ensure a reliable nitrogen separation. This will predominantly be realized by a conductivity controlled discharge of irrigation water. During the measurements the conductivity values were varied between 15 and 35 mS/cm and the corresponding irrigation water composition was analyzed (Table 5).

Table 5: Reservoir water composition and fluctuation range during both measurement periods

Parameter	Minimum	Maximum	Mean (n = 17)	Standard deviation
pH	3.7	6.4	5.4	0.9
LF in mS/cm	15.9	35.4	23.7	6.0
NH_4-N in mg/l	2,010	5,340	3,327	1,027
NO_2-N in mg/l	0	351	55[1)]	
NO_3-N in mg/l	988	2,735	1,846	545
N_{min} in mg/l	3,260	8,075	5,228	1,436

[1)] Detection in 3 from 17 samples only.

For an overall classification of the irrigation water into a “water hazardous class” in Germany the concentrations of different single compounds in the irrigation water are relevant which can be respectively ranged into a water hazardous class 1 (slightly hazardous to water), 2 (hazardous to water) or 3 (strongly hazardous to water). Relevant compounds in this respect are ammonium, nitrite, nitrate and sulfate.

Mixtures are presumed to be not hazardous to water if following requirements are fulfilled (VwVwS 1999):

- The content of compounds of water hazardous class 1 is less than 3 % by weight
- The content of compounds of water hazardous class 2 is less than 0.2 % by weight
- There are neither compounds added which can be attributed to water hazardous class 3 nor have carcinogenic effects or are of unknown character
- There is no addition of any dispersive agents.

The water analyses show that the threshold values of relevant concentrations for water hazardous class 1 compounds (3 % for ammonium sulfate) and water hazardous class 2 compounds (0.2 % for nitrites) were undercut clearly.

The sum of inorganic nitrogen compounds (NH_4-N, NO_2-N und NO_3-N) can be sufficiently calculated (correlation coefficient $R^2 = 0.98$) in a range between 15.9 and 35.4 mS/cm by measuring the electric conductivity with equation 1:

$$\sum N_{inorganic} = \sigma \cdot 0{,}22 \qquad \text{(Eq. 1)}$$

$N_{inorganic}$ = inorganic nitrogen compounds in g/l

σ = conductivity in mS/cm

In comparison to washing liquids of other DLG tested single stage trickling filters for pig keeping it becomes obvious that the nitrite concentrations were low in the bio filter irrigation water. In 14 of 17 samples only NH_4-N and NO_3-N were detected. The nitrogen content was around 4.4 g/l in samples with a conductivity value of 20 mS/cm and thereby higher compared to water of single stage trickling filters (Hahne et al. 2016).

Besides the nitrogen removal via water discharge an additional nitrogen removal is realized by the regular exchange of the filter material. In the latter nitrogen will be fixed as organic nitrogen and also accumulated as inorganic compounds (Figure 8). In these proper operated filters with the described nitrogen separation an algae growth occurs (Figure 10) which leads to a greening on the filter surface. In the frame of the investigations the algae growth did not cause any change in bio filter performance.

Figure 10: Algae growth on the wood chips layer by nitrogen accumulation

The useful life of the wood chips is depending on several factors as ammonia load, temperature, humidification, microbiological activity and the resultant pH values in the wood chips layer and in the water reservoir as well. On basis of the current state of research the maximum useful life of wood chips for the described application can be projected at 6 month.

Discussion

The advantage of the novel bio filter system consists in an extensive and reliable ammonia separation of at least 88 % compared to common bio filters. This will be ensured via pH control, a conductivity-controlled water discharge and a surplus irrigation of the bio filter material. Higher investment and running costs arise through this and the exchange of the filter material as well, which is required every six month. On the other hand the clearly reduced amount of discharge water of only 0.44 m^3 per fattening pig and year (Hahne 2016) is a cost-saving effect compared to biologically operating scrubbers (DLG 2014, DLG 2015). A comparison of total costs for exhaust air treatment in pig keeping shows that bio filters for small and middle livestock sizes up to 1,000 heads are more cost-efficient than trickling filters or multistage operations (Hahne 2016). If the novel bio filter system is despite of a higher effort still more cost-effective than other techniques, perspectives will arise especially for small and middle livestock sizes. This applies to new projects and the refitting of existing installations as well.

Conclusion

Bio filters, which are operated both with a pH control in a water reservoir and a surplus irrigation that secures a water saturation of the filter material and a regular reflow into the water reservoir, can be used for ammonia separation in pig keepings. At a maximum surface load of 440 $m^3/(m^2\ h)$ and proper moistened filter material ammonia separation of at least 88 % was achieved as long-term measurements show. For a secure and long-lasting operability a regular water discharge is indispensable to limit the conductivity at 25 mS/cm. Furthermore the wood chips layer has to be exchanged every six month due to the nitrogen accumulation in the filter material.

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