Potential ammonia volatilization from 39 different novel biobased fertilizers applied to soil – A laboratory study using European soils

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Abstract

Currently, the political focus on promoting a circular economy in the European Union is driving great interest in developing and using more biobased fertilizers (BBFs, most often waste or residue-derived). However, there have only been a few studies on ammonia (NH_3) volatilization from BBFs, e.g. regarding manure. This is despite the fact that NH_3 volatilization from agriculture results in major environmental impacts. Ammonia volatilization poses a risk to the environment and human health when entering the atmosphere, causing pollution in natural ecosystems through re-deposition and contributing to the formation of fine particulate matter (PM_x). In addition, since NH_3 volatilization results in the removal of plant-available N from agricultural systems, it constitutes an economic loss for farmers. This laboratory study assessed the potential NH_3 volatilization from 39 different BBFs commercially available on the European market. Moreover, the effect of incorporation, application rate, soil type, and soil moisture content was tested. Results showed a great variation between BBFs in potential NH_3 volatilization, both in terms of their temporal pattern of volatilization and amount of NH_3 volatilized. The potential NH_3 volatilization varied from 0 % of applied total N (olive oil compost) to 64 % of applied total N (manure and crop digestate) during a 27- or 44-day incubation period. Characteristics of BBFs (pH, NH_4^+-N, NO_3^--N, DM, C:N) and their interaction with time could explain 89 % of the variation in accumulated potential NH_3 volatilization. Incorporation of BBFs into soil effectively reduced potential NH_3 volatilization by 37 % to 96 % compared to surface application of BBFs. Potential NH_3 volatilization was not significantly affected by differences in application rate or soil moisture content, but varied between five different soils (with different clay and organic matter content), with the highest NH_3 volatilization potential from Acidic sandy soil.

Keywords: (Potential) ammonia volatilization, Biobased fertilizers, Incorporation, Surface application, Incubation, Soil type.

1 Introduction

Biobased fertilizers (BBFs) can be defined as materials or products derived from biomaterials (plant, animal or microbial origin, often wastes, residues or side-streams from agriculture, industry or society) with a content of bioavailable plant nutrients suitable to serve as a fertilizer for crops. Producing and using BBFs is therefore a means to recycle otherwise potentially wasted nutrients. They can replace mineral fertilizers, lowering the environmental and climate impacts of mineral nitrogen (N) fertilizer production, and the environmental and resource depletion impacts of phosphorus (P) and potassium (K) mining and extraction.

Therefore, there is a lot of political interest in promoting waste- or residue-based BBFs in a circular economy context in the European Union (EU) (EC, 2015). A new EU Fertilizing Products Regulation (EU, 2019/1009) has been approved by the European Commission and will be implemented in the member states in the coming years. This is expected to lead to an increase in the use of novel BBFs, as the new fertilizer regulation will allow various BBFs to enter the free trade market for fertilizers, provided they comply with certain production and quality standards (Schoumans et al., 2019). The recent instability of mineral fertilizer markets, with rapidly fluctuating fertilizer prices (Baffes and Koh, 2021), will also increase interest in alternative and less energy market-sensitive nutrient sources.

However, knowledge about the N fertilizer effect and environmental impacts of the use of novel BBFs is currently very limited. An important aspect is ammonia (NH_3) volatilization, which is of growing environmental concern (Li et al., 2020). Globally, 80 % of NH_3 volatilization is associated with human activities, mainly related to animal husbandry and the application of fertilizers (UNEP, 2019).

Once volatilized, NH_3 reacts with acidic gasses which impacts the formation, transformation, and deposition of aerosols (Akiyama et al., 2004). Re-deposition of NH_3 may contribute directly or indirectly to soil acidification, eutrophication of aquatic environments, and biodiversity loss. Furthermore, NH_3 may be oxidized to nitrate (NO_3^-) and further transformed to the potent greenhouse gas N_2O through denitrification (Ferm, 1998). In addition to environmental concerns, secondary ultrafine particles in the atmosphere formed by NH_3 and other reactive compounds pose a significant human health risk (Sanz-Cobena et al., 2014). Moreover, NH_3 volatilization may also reduce the inorganic N content in the soil applied with BBFs, thereby causing decreases in yields, with consequential economic losses for the farmer (Kirchmann and Lundvall, 1993).

Theoretically, BBFs with a high pH and/or high ammonium (NH_4^+) content will be expected to have higher NH_3 volatilization potential compared to BBFs with a low pH and/or low NH_4^+ content (Sommer and Feilberg, 2013). However, BBFs with a low NH_4^+ content may contain a substantial pool of easily mineralizable organic N, which upon soil microbial decomposition of the BBF will become mineralized and provide NH_4^+-N, increasing the potential for NH_3 volatilization. Moreover, the physical form, e.g. pelletized, liquid or powder, may affect the NH_3 volatilization potential, as it affects the dissolution and diffusion of the NH_4^+ content of the BBF into the soil (Sommer et al., 2004). NH_3 may also be lost during the BBF production process, as observed e.g. during the composting (Usmani et al., 2020) and drying of anaerobic digestate (Awiszus et al., 2018).

Apart from the BBF properties, the soil type to which the BBF is applied may affect the NH_3 volatilization potential. In a study including 22 soils differing in properties, Duan and Xiao (2000) found

NH_3 volatilization rates to be positively correlated with soil pH, $CaCO_3$, and salt content and negatively correlated with soil organic matter (SOM) content, cation exchange capacity (CEC), and clay content. In acidic soils, or soils with a low content of pH buffer components, NH_3 volatilization will rapidly decline (Sommer et al., 2004). Moreover, soil moisture may affect NH_3 volatilization potential, as the concentration of NH_4^+ content in solution is lower at higher soil moisture, which may lead to lower NH_3 volatilization (Haynes and Sherlock, 1986).

Previous studies on NH_3 volatilization from BBFs have focused on a limited variety and number of BBFs, including mainly manure (Akiyama et al. (2004), Bernal and Kirchmann (1992), Mkhabela et al. (2006)) and to some extent digestates (Nkoa (2014), Verdi et al. (2019)). Ammonia volatilization from both livestock and green manure has been shown to be generally higher compared to mineral N fertilizers (Ma et al., 2021). Therefore, studies on NH_3 volatilization from novel BBFs covering a wide range of source material as well as chemical and physical properties are strongly needed.

Thus, the overall objective of this study was to determine the potential NH_3 volatilization from the use of 39 different BBFs under variation of soil type and moisture conditions, as well as application rate and method, and to derive suggestions for the optimal application conditions under practical settings.

We wanted to test the following hypotheses: i) Novel BBFs are expected to behave in the same way as more traditional BBFs, e.g. manure, where a high pH and/or high NH_4^+ content will lead to high NH_3 volatilization potentials; ii) Biobased fertilizers produced partly or fully from poultry manures are expected to have high NH_3 volatilization potentials, due to their high content of uric acid, rapidly hydrolyzing to NH_4^+; iii) Application of BBFs to soils with a high pH will increase their NH_3 volatilization potential, whereas soils with a high organic matter (OM) content, high clay content, and/or high CEC will decrease potential NH_3 volatilization of BBFs; and iv) Incorporation of BBFs into soil or sand will decrease the potential NH_3 volatilization.

The method used in this study was developed as a standard test method for the EU H2020 LEX4BIO project. The method is meant to be used as an assessment of whether the BBF poses a risk of NH_3 loss. It allows estimation of the potential NH_3 loss from a BBF under conditions of constant removal of NH_3

and CO_2, creating a maximum gradient for both gasses because they interact in enhancing NH_3 volatilization (Husted et al., 1991). These conditions simulate field conditions with high wind speeds and no vegetation cover. Moreover, to assess the NH_3 volatilization potential from the 39 BBFs independently from their interactions with the soil, they were also applied to pure sand, imitating the physical properties of soil, but lacking the chemical and biological characteristics that may affect NH_3 volatilization.

2 Materials and methods

2.1 Materials

2.1.1 Biobased fertilizers

39 BBFs, produced from various waste- and side streams of agricultural, urban or industrial origin, were included in this study. All BBFs were available on the European market or on regional/national markets at the time of writing. A description of raw materials and technologies used, along with product function categories (PFC) and component material categories (CMC) according to the new EU Fertilizing Products Regulation (EU, 2019/1009), is provided in table 1. The CMCs refer to the processing of BBFs, e.g. compost and digestate and the input material, e.g. industry by-products. The PFCs are related to the main BBF functions; subcategories relate to whether it is liquid or solid and the amount of mineral and organic components it contains.

Moreover, the BBFs in this study were also grouped based on more classical terms used in the literature (Table 1). Standard categories were digestate and compost. Meat and bone meal and other products from the meat industry were grouped together as animal by-products. Struvites and other BBFs derived from mineral extractions were grouped together as mineral precipitates. Potato fruit juice and other BBFs produced solely from plant materials were grouped together as plant-based BBFs. BBFs in a pelletized form produced from various materials were lumped in a group called “mixed”.

Before application to soil or sand, all BBFs were homogenized. For this purpose, non-fluid BBFs were carefully crushed using a mortar until they could pass through a 2-mm sieve. For BBFs with a high-fiber content, which could not be crushed using the mortar, fibers were cut with scissors and passed through a 2-mm sieve. Dry BBFs with particle sizes < 2 mm and homogenous liquid BBFs were not further processed. Non-homogenous liquid and moist BBFs, e.g. digestates and composts, were homogenized using a blender for approximately one minute. Comparisons of NH_3 volatilization potential from homogenized and non-homogenized BBFs are included in Appendix A, Fig. A1.

Table 1: Raw material(s) and technologies used in production, along with their product function category (PFC) and component material categories (CMC) (EU, 2019/1009), and grouping of the biobased fertilizers (BBFs) included in this study. All the BBFs are presented here with their acronyms (three letters/numbers) Full product names and manufacturers are shown in the supplementary material (Table A1).

BBF	Raw material	Technology	PFC**	CMC*	Group
ASL	Product of nutrient-recycle-plant	Anaerobic digestion	1 A-II	4	Mineral precipitate
AV4	Broiler litter	Drying, granulating, and pelletizing	1 A-I	10	Poultry manure
AV8	Broiler litter, blood meal, and potassium sulfate	Drying, granulating, and pelletizing	1 B-I	10	Poultry manure
BA6	Plant-based residues (wheat and maize)	Fermentation and distillation	1 A-II	4(6)***	Plant-based
BIH	Animal horns	Pelletizing (Sphero technology)	1 A-I	10	Animal by-product
BIL	Broiler litter and seaweed	Drying and pelletizing	1 A-I	10	Poultry manure
BIO	Meat and bone meal, apatite, vinasse, poultry manure, and potassium sulfate	Pelletizing	1 B-I	10	Animal by-product
BIP	Different plant wastes, e.g. leftovers from sugar production	Liquidization	1 A-II	6	Plant-based
BLM	Blood meal		1 A-I	10	Animal by-product
BO1	Potato cell water	Evaporation	1 A-II	6	Plant-based
BO2	Molasses	Anaerobic digestion	1 A-II	6	Plant-based
BO4	Vinasse (sugar production)	Anaerobic digestion	1 A-II	6	Plant-based
BVC	Municipal organic food waste	Anaerobic digestion and composting	1 A-I	3	Compost

CGR	Wastewater supernatant	Struvite precipitation	1 C-I	12	Mineral precipitate
ECO	Blood and feather meal	Pelletizing	1 B-I	10	Animal by-product
FEK	Poultry manure	Drying and processing (extrusion process)	1 A-I	10	Poultry manure
FEL	Poultry manure	Drying in low temperature and pelletizing	1 A-I	10	Poultry manure
GRF	Manure and crop digestate	Digestion	1 A-II	5	Digestate
HDG	75 % slurry, 25% source-separated Organic household waste plus organic industrial wastes	Digestion	1 A-II	5	Digestate
ILF	Shavings of treated hides and skins	Enzymatic hydrolysis	1 A-II	10	Animal by-product
MAL	Mixture of malt germ, malt, minerals, and vinasse	Drying and pelletizing	1 A-I	6	Plant-based
MB2	Meat and bone meal	Pelletizing (Sphero technology)	1 B-I	10	Animal by-product
MO13	Feather meal	Pelletizing	1 A-I	10	Animal by-product
NAD	Liquid manure and vegetables residue	Fermentation, drying, and pelletizing	1 B-I	4	Mixed
NE4	Sugar, molasses, syrup, mycelium from *Aspergillus niger*	Drying and pelletizing	1 A-I	6	Plant-based
NE7	Plant-based organic raw material and crude phosphate, *Bacillus amyloliquefaciens*	Drying and pelletizing	1 B-I	6	Plant-based
OG1	Meat and bone meal	Pelletizing	1 B-I	10	Animal by-product
OG2	Horn meal (pig bristles)	Hydrolysis	1 A-I	10	Animal by-product
OOC	Olive oil production residues	Composting	1 A-I	3	Compost
OPU	Poultry manure	Pelletizing	1 B-I	10	Poultry manure
PAL	Fermented biochar and high-quality clay and rock flour	Pyrolysis and fermentation "Terra Preta"	1 A-I	4	Plant-based
PCS	Sewage water	P extraction via struvite	1 C-I	12	Mineral precipitate
PCW	Potato cell water	Evaporation	1 B-II	6	Plant-based
PRI	Organic material animal and/or vegetal origin + mineral granules	Compost into granules, then blending with mineral granules	1 B-I	3(6)***	Mixed
SDG	Agro and food waste + seaweed	Digestion	1 A-II	6	Digestate
SIF	Animal and vegetal raw materials	Granulating	1 A-I	6(10)***	Mixed

SYS	Ammonium-nitrogen and potassium	Digestion using reverse osmosis	1 C-II	6	Mineral precipitate
TRS	Fish soluble, 78 % proteins, 16 % ash	Enzymatic hydrolysis	1 A-I	10	Animal by-product
VEC	Residues of the digestion of horse manure	Composting	1 A-I	3	Compost

* CMC 3: Compost, CMC 4: Fresh crop digestate, CMC 5: Digestate other than fresh crop digestate, CMC 6: Food industry by-products, CMC 10: Derived products within the meaning of Animal By-products Regulation, and CMC 12: Precipitated phosphate salts and derivates (EU, 2019/1009). ** PFC 1 A-I: Solid organic fertilizer: ≥ 15 % organic C, PFC 1 A-II: Liquid organic fertilizer: ≥ 5 % organic C, PFC 1 B-I: Solid organo-mineral fertilizer: ≥ 7.5 % organic C, 1 B-II: Liquid organo-mineral fertilizer: ≥ 3 % organic C, 1 C-I: Straight solid inorganic macronutrient fertilizer, and 1 C-I: Compound solid inorganic macronutrient fertilizer (EU, 2019/1009). *** For BBFs which belong to more than one CMC group, the secondary CMC group is included in brackets.

2.1.2 Soil and sand matrix

For the incubation experiments on potential NH_3 volatilization, we used either pure sand or different soils as the matrix to which the BBFs were added. The sand used was in the size range 0.4 – 0.9 mm, contained 0.2 % OM, and had been washed with water (Dansand A/S, Brædstrup, Denmark). Five different soils were used in the different experimental runs. These soils were selected from the experimental field sites of the EU H2020 LEX4BIO project, representing some of the variation in soil types present in Europe (Table 2). The soil samples were collected from the top 0-30 cm. Acidic sandy soil was air-dried and sieved to 2 mm. Acidic clay soil, Alkaline loamy soil, Neutral loamy soil, and Alkaline clay soil were sieved to 4 mm and air-dried.

Table 2: Soil characteristics of the soils used in the experiment. Values are means ± Standard error. For N, C, pH Milli-Q water, pH $CaCl_2$, water-holding capacity (WHC), and cation exchange capacity (CEC), N = 3, except WHC for Alkaline loamy soil, where N = 2. For organic matter (OM), clay, silt, fine sand, and coarse sand, N = 1.

	Acidic sandy soil	**Acidic clay soil**	**Neutral loamy soil**	**Alkaline loamy soil**	**Alkaline clay soil**

Soil geography	Eastern Denmark	Southern Finland	Southern Germany	Southern France	Southern Spain
Coordinates	55°40'28.6"N 12°17'17.8"E	60°48'15.6"N 23°27'06.5"E	48°42'53.8"N 9°12'52.2"E	43°29'35.6"N 1°12'07.6"E	37°24'06.6"N 5°35'45.9"W
N (%)*	0.16±<0.01	0.27±<0.01	0.19±<0.01	0.12±<0.01	0.16±0.01
C (%)*	1.39±0.01	2.89±0.02	1.31±<0.01	0.83±0.03	4.07±0.03
pH Milli-Q water	6.64±0.07	6.79±0.03	7.75±0.01	7.89±0.01	8.35±0.02
pH $CaCl_2$	5.73±0.11	5.74±0.01	7.04±0.02	7.20±0.05	7.66±0.01
WHC (% of dry mass)	35.5±0.3	46.8±0.4	41.4±1.0	39.1±1.7	46.7±0.3
CEC_{pH7} (cmol/kg)**	7.1±0.4	15.4±4.0	10.4±3.1	11.0±0.7	34.9±4.6
OM (%)***	2.1	4.8	2.2	1.3	1.9
Clay <0.002 mm (%)***	9.6	48.8	33.2	14.2	61.7
Silt 0.002-0.02 mm (%)***	8.4	26.1	30.2	20.9	25.9
Fine sand 0.02-0.2 mm (%)***	44.7	14.5	33.0	47.7	9.2
Coarse sand 0.2-2 mm (%)***	35.2	5.9	1.4	15.9	1.4

* Total C and N was determined by Dumas combustion. ** CEC determined by the NH_4^+ acetate method, quantifying NH_4^+-N with flow injection analysis. *** Texture and OM content determined at Agrolab Sarstedt by sieving and sedimentation for texture and by Dumas combustion for total OM.

2.2 Incubation setup for determining potential ammonia volatilization

The experiment was performed using a static diffusion enclosure method, developed by modifying the procedures described by the following authors (Husted et al., 1991; Ndegwa et al., 2009; Mandal et al., 2016). A detailed methods description is openly available online in ERDA, the repository of the University of Copenhagen:

https://erda.ku.dk/archives/5ee73487bccba9e23180f0bacbdb4d62/published-archive.html.

One hundred g (oven dry weight) of air-dry sieved soil/sand was added to 120 mL plastic containers. The soil was compacted to 1.25 g cm^{-3} (Acidic sandy soil and Alkaline loamy soil), 1.11 g cm^{-3} (Neutral loamy soil and Alkaline clay soil), and 1 g cm^{-3} (Acidic clay soil). The sand was not compacted. Milli-Q water was added to each container with a pipette to adjust the water content to 60 % of the soil's water-holding capacity (WHC, determined modified according to ISO 14238). For the sand, it was assessed that 20 mL of Milli-Q water was suitable for 100 g (dry weight) of sand. The moisture content of the air-dry soil/sand and the water content of the BBFs were subtracted from the amount of water to be added to ensure an equal moisture content in all setups. The containers with moist soil/sand were pre-incubated for five days at 15 °C in a dark incubator (Termaks, A/S Ninolab, Solrød, Denmark) before addition of the BBF.

To mimic areas of a field receiving high BBF applications on its soil surface, e.g. a strip of liquid digestate from a trailing hose or the area around a solid BBF pellet, 400 kg total-N ha^{-1}, corresponding to 0.004 g total-N cm^{-2}, was applied as the standard rate for all 39 BBFs in experiment 1 (Table 3). In a different trial, eight selected BBFs were either surface applied and distributed evenly on the soil/sand surface (standard application method) or they were applied and thoroughly incorporated by mixing into the soil/sand before addition of Milli-Q water (see table 3, experiment 2). Furthermore, to test the implication of the application rate, selected BBFs were applied at both the standard rate (1* application rate), which corresponds to 720 mg N kg^{-1} soil; half the rate (½ * application rate), corresponding to 360 mg N kg^{-1} soil (200 kg BBF N ha^{-1}); and double the rate (2* application rate), corresponding to 1440 mg N kg^{-1} soil (800 kg BBF N ha^{-1}) (see table 3, experiment 3). The effect of the soil type was tested with five different BBFs (see table 3, experiment 4). Moreover, the effect of the soil moisture was tested with three BBFs (see table 3, experiment 5).

Table 3: Overview of treatments in the experiment.

Experiment	**No of BBFs**	**Soil/sand**	**BBF application**	**Water content** (% of WHC)	**BBF application rate** (mg N kg^{-1} soil)
1	39	Sand	Surface	60	720
2	8	Acidic sandy soil; Sand	Surface; Incorporated	60	720
3	3	Acidic sandy soil	Surface	60	360; 720; 1440
4	5	Acidic sandy soil; Acidic clay soil; Neutral loamy soil; Alkaline loamy soil; Alkaline clay soil	Surface	60	720
5	3	Acidic sandy soil	Surface	30; 60; 90	720

Three replicates of each treatment were included in all experimental runs. Three replicates of controls with only sand/soil and no fertilizer were also included, as well as two blank tests of the setup without any sand/soil or fertilizer. Both no-treatment controls and blanks generally had NH_3 concentrations below the detection limit and were therefore not accounted for. Moreover, a reference fertilizer of ammonium bicarbonate (NH_4HCO_3) dissolved in Milli-Q water was included in each experimental run. This was, firstly, to serve as a reference for maximum potential NH_3 loss (due to all N being present as NH_4^+ and bicarbonate promoting pH rise upon application) and, secondly, to allow comparisons between experimental runs.

Each 120 mL plastic container with soil/sand and applied BBF, surface area of 18 cm^2, was incubated in a glass canning jar (0.75 L) with an airtight lid. A trap consisting of 5 mL 0.2 M sulfuric acid (H_2SO_4) in an open plastic container (26 mL airtight plastic container, Frisenette ApS, Knebel, Denmark) was placed in the canning jar. The amount and concentration of H_2SO_4 was calculated stoichiometrically so that the H_2SO_4 was able to trap the double amount of NH_3 that could potentially volatilize (Ndegwa et al., 2009). The surface area of the acid in the acid trap was 7 cm^2. In a pre-trial, the efficiency of the acid trap was tested with NH_4HCO_3 added to sand. The result showed that 82 % of the N added as NH_4HCO_3 was recovered in the acid trap during an experimental period of 44 days (Fig. 1). This is within the range of previously reported NH_3 recovery values of 73 – 103 % in laboratory setups (Woodward et al., 2011) and indicates that the setup did not have any significant leakage. The trap collected after the last trap exchange in the test trial with NH_4HCO_3 contained NH_3 and, thus, NH_3 was still volatilizing at the end of the trial period. If the experimental period had been extended, the recovery percentage would likely have approached 100 %.

A base trap consisting of 10 mL of 1 M sodium hydroxide (NaOH) in a plastic shot glass was additionally inserted in the canning jar to trap any CO_2 evolved during the incubation. Furthermore, a plastic shot glass containing 10 mL of deionized water was placed in the canning jar to maintain high humidity and prevent desiccation of the BBF and soil sample over time.

Canning jars were placed in a dark incubator (Termaks, A/S Ninolab, Solrød, Denmark) set to 15 °C to reflect a mean European air temperature.

Trap exchange and quantification of NH_3

The H_2SO_4 and NaOH traps were exchanged 1, 2, 5, 9, 14, 20, and 27 days after the start of the experiment at the same time of day ± 30 minutes. For the experimental run with all BBFs surface-applied to sand (Experiment 1 table 3), two additional sampling times (35 and 44 days after the start) were applied. However, four of the 39 BBFs (MAL, BLM, ASL, and GRF) were included in a different experiment which only ran for 27 days. The base traps with NaOH were replaced, but not analyzed for CO_2 content. The acid traps with H_2SO_4 were stored at ambient temperature with air-tight lids for a maximum of two weeks until analysis of the NH_4^+-N content. The stability of the NH_4^+ content in the traps was assessed to be at least three weeks by analyzing the NH_4^+-N in the same traps at different time points. The NH_3 trapped in the H_2SO_4 was quantified by analyzing the NH_4^+-N content in the traps using flow injection analysis (FIAstar 5000 flow injection analyser (Foss Analytical, Hillerød, Denmark)). The lowest detected concentration was 0.0045 mg NH_4^+-N L^{-1}. This method of quantifying NH_3 was tested against the titration method (see Fig. A2).

2.3 Analysis of soil and BBFs

At the end of each experimental run, all samples of soil/sand with BBFs applied were mixed thoroughly and a subsample of five g was collected to determine the pH value. The pH was determined on the last day of the experiment (day 27 or day 44) as 1:5 in Milli-Q water.

Dry matter (DM) of solid BBFs was determined at 105°C by TGA701 (Leco) and that of liquid BBFs by drying in a ventilated oven (105°C) until the weight did not change. The total C and N of BBFs was determined by the Dumas dry combustion method (Trumac or CHN628, Leco). The water-soluble NH_4^+-N and NO_3^--N of the BBFs were analyzed in 1:60 water extracts with a continuous flow analyzer (Skalar San ++ System). All analyses of BBFs were carried out on their original dry matter content.

2.4 Statistics

All illustrations were made in SigmaPlot 14.00 (Systat Software, Inc.). All statistical analyses were performed in R version 4.0.2 (R Core Team). A minimum significance level of $p<0.05$ was applied for all tests. Data were visually assessed with diagnostic plots to check the homogeneity of variance and data were square-root transformed for all statistical analyses due to heterogeneity of variance.

The analyses were performed on the accumulated NH_3-N volatilized as a percentage of total added N for all days. The repeated measurements of accumulated NH_3 volatilization of each sample were accounted for by setting the sampling day as a repeated variable. This transformation was applied for all statistical analyses. All analyses were performed using linear mixed models and pairwise comparisons of least square means. R^2 values and correlation coefficients were retrieved from the linear mixed models. A quadratic term of measurement day was included in the linear mixed models to account for non-linearity. Moreover, the sample number was always included as a random effect. BBF was included as a random effect for experiment 1 (table 3) and for the correlation coefficients and R^2 value for experiment 4 (table 3), but not for the remaining analyses, as these were performed separately for each BBF treatment. See table A2 for further information on statistics.

3 Results

3.1 BBF properties

The properties of the 39 BBFs varied substantially (Table 4). The greatest variations were found in NH_4^+-N content and dry matter (DM) content.

Table 4: Properties of the 39 BBFs included pH (1:5 in Milli-Q water), total N, NH_4^+-N, NO_3^--N, Dry-matter (DM) content, total C, and C:N ratio. N, DM, and C are reported as % of fresh weight (FW). Values are means, N=2, except for C and N of liquid BBFs where N=5 and pH where N = 3.

	pH	**N** (% of FW)	**NH_4^+-N** (% of total N)	**NO_3^--N** (% of total N)	**DM** (g kg^{-1} of FW)	**C** (% of FW)	**C/N**
ASL	7.42	4.76	100	<0.1	216	0.11	0.02
AV4	6.27	3.98	25	<0.1	893	38.9	9.77
AV8	6.51	7.24	8.4	<0.1	920	38.9	5.38
BA6	4.85	5.57	1.4	<0.1	907	43.6	7.83
BIH	6.75	13.8	2.0	<0.1	892	42.5	3.08
BIL	6.32	4.11	19	0.4	894	38.9	9.47
BIO	5.69	7.39	3.5	<0.1	941	35.9	4.85
BIP	5.21	6.50	49	0.7	569	21.0	3.24
BLM	6.85	14.7	0.2	<0.1	917	49.6	3.37
BO1	5.88	1.46	20	<0.1	184	7.53	5.16
BO2	6.59	1.66	0.9	0.9	521	20.5	12.3
BO4	6.23	3.56	1.3	0.6	620	21.2	5.96
BVC	8.56	1.57	8.1	<0.1	557	14.7	9.37
CGR	8.38	5.53	0.7	<0.1	605	0.20	0.04
ECO	5.48	11.6	2.7	<0.1	892	45.3	3.89
FEK	6.43	3.94	20	<0.1	901	34.7	8.81
FEL	6.70	4.51	3.3	<0.1	925	36.0	7.98
GRF	8.19	0.37	75	0.3	45	2.02	5.46
HDG	8.15	0.44	61	0.1	46	1.57	3.57
ILF	6.37	8.84	2.1	<0.1	561	25.1	2.83
MAL	5.02	4.37	28	<0.1	955	35.0	8.02
MB2	6.39	8.01	0.6	<0.1	959	35.6	4.45
MO13	5.07	14.2	1.0	<0.1	927	49.0	3.45
NAD	8.43	2.49	0.4	3.6	881	37.8	15.2
NE4	8.41	3.99	0.2	<0.1	925	24.8	6.22
NE7	6.51	6.65	1.7	<0.1	936	35.9	5.39
OG1	5.73	10.2	0.7	<0.1	949	43.4	4.26
OG2	5.29	13.9	1.0	<0.1	940	48.3	3.47
OOC	8.13	1.24	0.3	<0.1	900	40.8	32.9
OPU	8.44	2.69	11	<0.1	893	33.3	12.4
PAL	5.55	4.89	19	0.1	907	38.8	7.92
PCS	9.21	5.61	1.1	<0.1	614	0.27	0.05
PCW	4.70	1.53	15	1.1	339	11.2	7.29
PRI	5.80	17.4	51	47	968	5.46	0.31
SDG	8.40	0.41	68	<0.1	38	1.08	2.63
SIF	5.87	9.92	4.3	0.3	950	42.0	4.23
SYS	7.06	8.07	95	0.5	384	0.12	0.01
TRS	5.87	13.6	5.0	<0.1	975	37.2	2.74
VEC	8.00	0.59	0.3	3.7	262	7.76	13.2

3.2 Experiment 1: Potential NH_3 volatilization from all BBFs

The 39 BBFs surface applied to sand varied greatly in terms of accumulated potential NH_3 volatilized at the end of the incubation period (Fig. 1). However, all the BBFs showed some potential NH_3 volatilization during the incubation period, except for OOC (data not shown), where volatilization was below the detection limit on all days. Moreover, the temporal pattern of potential NH_3 volatilization during the incubation period differed between BBFs. Some BBFs had a high initial potential NH_3 volatilization and had concave shaped curves of accumulated potential NH_3 volatilization. For other BBFs, however, the initial potential NH_3 volatilization was very low and increased towards the end of the incubation period, resulting in convex curves of accumulated potential NH_3 volatilization.

[Insert Fig. 1 around here, 2-column fitting image]

Among the six CMCs, the accumulated NH_3 volatilization on day 27 was lowest for BBFs in CMC 12, followed by CMC 3 < 4 < 6 < 10 < 5. Among the six PFCs, the accumulated NH_3 volatilization on day 27 was lowest for BBFs in PFC 1 A-II, followed by 1 C-I < 1 C-II < 1 B-II < 1 A-I < 1 B-I < 1 A-II. Among the seven BBF groups, the accumulated NH_3 volatilization on day 27 was lowest for Compost followed by Mineral precipitate < Mixed < Plant based < Animal by-products < Poultry manure < Digestate.

Based on the number of significant differences between categories/groups in the three different categorizations included, CMCs and PFCs were not as good at predicting accumulated potential NH_3 volatilization compared to the groups based on traditional categorizations (Table 5).

Table 5: BBF groups, CMCs, and PFCs which were significantly different from each other based on a linear mixed model including all time points. No. of groups = 7, no. of CMCs = 6, and no of PFCs = 6.

Group, CMC, and PFC			p-value	Estimates of least square means*
Animal by-product	≠	Digestate	0.002	-3.46
Animal by-product	≠	Mineral extract	0.018	2.16
Compost	≠	Digestate	0.002	-3.19

Compost	≠	Mineral extract	0.031	2.43
Digestate	≠	Mineral extract	<0.001	5.62
Digestate	≠	Mixed	0.014	3
Digestate	≠	Plant based	0.002	3.13
Digestate	≠	Poultry manure	0.009	2.76
Mineral extract	≠	Mixed	0.010	-2.62
Mineral extract	≠	Plant based	0.003	-2.49
Mineral extract	≠	Poultry manure	0.0004	-2.87
CMC 5	≠	CMC 3	0.007	-3.95
CMC 5	≠	CMC 4	0.002	-3.65
CMC 5	≠	CMC 6	0.037	2.47
CMC 5	≠	CMC 10	0.024	-2.89
PFC 1 C-II	≠	PFC 1 A-I	0.033	3.58
PFC 1 C-II	≠	PFC 1 B-I	0.034	3.75

* Estimates of differences in least square means are square-root transformed.

A linear mixed model including BBF properties (pH, NH_4^+-N, NO_3^--N, DM, C:N) and their interaction with time explained 89 % of the variation in accumulated potential NH_3 volatilization during the experimental period of 44 days. Potential NH_3 volatilization was positively correlated with $pH_{\text{Milli-Q water}}$ (0.312) and NH_4^+-N content (0.013) and negatively correlated with NO_3^--N content (-0.007), DM content (-0.01), and C:N ratio (-0.002).

3.3 Experiment 2: Soil vs. sand and BBF application technique

The potential NH_3 volatilization was significantly lower for soil (Acidic sandy soil) compared to sand for the NH_4HCO_3 reference and all BBFs, except BO1 ($P<0.0001$) (Fig. 2). Moreover, potential NH_3 volatilization was significantly lower for incorporation compared to surface application for all BBFs ($P<0.0001$), except BO1 (Fig. 2). For the digestate HDG, the NH_3 volatilization after one day was reduced by 88 % as a result of incorporation into soil relative to surface application. For the remaining BBFs, no noteworthy NH_3 had volatilized during the first day of incorporation. Looking at the accumulated NH_3 volatilization after 27 days of incubation, incorporation into soil reduced NH_3 volatilization by 37 % and 64 % for the NH_4HCO_3 reference and HDG respectively compared to surface application on soil. For AV4, SIF, PAL, ECO, NAD, and BVC, incorporation reduced NH_3 volatilization by more than 96 %.

[Insert Fig. 2 around here, 2-column fitting image]

3.4 Experiment 3: BBF application rate

For the NH_4HCO_3 reference and AV4 (Fig. 3 A+B), the accumulated NH_3 volatilized was approximately doubled at the end of the incubation (day 27) when doubling the application rate. For ECO and HDG (Fig. 3 C+D), the differences between application rates were smaller, but showed a similar trend. Therefore, the potential NH_3 volatilized did not generally increase linearly with an increasing application rate. However, there were no overall significant differences between the three application rates. Only ½ the application rate was significantly different from both 1 and 2 times the application rate for AV4 and the NH_4HCO_3 reference (Fig. 3 A and B).

[Insert Fig. 3 around here, 2-column fitting image]

3.5 Experiment 4: Effect of soil type

The accumulated potential NH_3 volatilization was significantly higher for Acidic sandy soil for all BBFs and the NH_4HCO_3 reference at the end of the incubation period compared to Acidic clay soil, Neutral loamy soil, and Alkaline clay soil (Fig. 4). The accumulated potential NH_3 volatilization was lowest for Acidic clay soil and Alkaline clay soil.

[Insert Fig. 4 around here, 2-column fitting image]

In a linear mixed model, soil characteristics (pH_{CaCl2}, OM, and clay content) and their interactions with time explained 88 % of the variation in potential NH_3 volatilization for all days, all five soils and all

BBFs + NH_4HCO_3 reference. Accumulated potential NH_3 volatilization was negatively correlated with pH_{CaCl2} (-0.15), OM (-0.13), and clay content (-0.002).

3.6 Experiment 5: Effect of soil moisture

No clear pattern across BBFs was observed for the differences in soil moisture (WHC levels) (Fig. 5). There were no marked differences in NH_3 volatilization between WHC levels for ECO and HDG. For AV4, 90 % WHC resulted in the highest volatilization and 30 % WHC in the lowest; whereas, the opposite was true for the NH_4HCO_3 reference.

[Insert Fig. 5 around here, 2-column fitting image]

4 Discussion

4.1 Experiment 1: Potential NH_3 volatilization from biobased fertilizers

The great variability in the quantity and temporal pattern of potential NH_3 volatilization from the 39 BBFs included in this study highlights the importance of accounting for the fertilizer type when predicting NH_3 volatilization upon application.

4.1.1 Temporal pattern in NH_3 volatilization

The Michaelis-Menten or Monod type equation has often been used to describe NH_3 volatilization from soil upon application of slurry (Monaco et al., 2012). Liao et al. (2019) observed high initial NH_3 volatilization during the first 2-3 days from digestates applied to soil using a dynamic flow-through setup. After the initial days, NH_3 volatilization was very low. Thus, the pattern of NH_3 volatilization followed a concave/Michaelis-Menten type equation. The findings of Liao et al. (2019) are in agreement with this study regarding the three digestates SDG, GRF, and HDG (Fig. 1). However, for the majority of BBFs, the pattern of potential NH_3 volatilized followed a convex shape in the initial period of 20 days, where a lag phase in the potential NH_3 volatilization was observed (Fig. 1). This is in agreement with the findings of Erwiha et al. (2020), who also found convex shaped curves of accumulated NH_3 volatilization during the initial 2-3 weeks after application of blood meal and feather meal on a fine clay loam in a field trial in Colorado, USA. This convex curve of NH_3 volatilization potential could indicate that NH_3 volatilization from the majority of BBFs is controlled by the rate of mineralization. This is supported by the observation that nearly all samples with BBFs surface applied to sand and thoroughly mixed at the end of the incubation period (day 27/44) had a pH above 7 (Table A3). Only SYS had a pH below 7 (6.7) and only three treatments (ASL, MAL, and SYS) had a pH below the no treatment control of pH 7.9. Thus, pH was not the factor limiting NH_3 volatilization, at least not at this last stage, where close to all NH_4^+-N could potentially volatilize as NH_3.

4.1.2 BBF properties' ability to predict NH_3 volatilization potential

89 % of the variation in accumulated NH_3 volatilization potential could be explained by initial BBF properties (pH, NH_4^+-N, NO_3^--N, DM, and C:N) and their interaction with time. The ammonium-N content and pH of the BBFs showed a weak positive correlation with potential NH_3 volatilization on the first day. This confirms our hypothesis that BBFs with a high NH_4^+-N content and/or high pH will have a high NH_3 volatilization potential.

4.1.3 Digestates

Generally, digestates with animal manure and slurry as source material have a higher NH_3 volatilization potential upon application in the field compared to non-digested animal manure and slurry (Moeller and Stinner, 2009; Nkoa, 2014). In this study, the three digestates (HDG, SDG, and GRF) (Table 1) were also the BBFs with the highest NH_3 volatilization potential. Rabiger et al. (2020) found NH_3 volatilization of 7.6-18.3 % of total applied N from digestate applied at five different sites in Germany, using a standardized dynamic closed chamber method. Our results of NH_3 volatilization from digestates surface applied to sand were higher (39-64% of applied N). However, the results of Rabiger et al. (2020)

are comparable, although slightly higher, compared to the results in this study on NH_3 volatilization potential from the digestate HDG surface applied to five different soils (1.2-15% of applied N).

4.1.4 Poultry manure containing BBFs

This study included six BBFs containing poultry manure or poultry litter (Table 1). In a previous laboratory incubation study, using a sandy loam soil, NH_3 volatilization from application of poultry manure was found to range from 3.2% - 6.1% of applied N (Akiyama et al., 2004). For AV4 surface applied to the Acidic sandy soil, accumulated NH_3 volatilization on day 27 was only 4.1 % of applied N, which is comparable to the results of Akiyama et al. (2004). Our hypothesis that BBFs produced from poultry manure would have a high NH_3 volatilization potential can neither be completely confirmed nor completely rejected, as the poultry manure BBFs were the group with the second highest accumulated NH_3 volatilization potential after digestates, but very similar to the animal by-product BBFs (see Fig. A4). The comparatively low NH_3 volatilization potential may be due to the production process of the BBFs containing poultry manure, since this typically includes a drying step and thereby already promotes a potential NH_3 loss during production. Lockyer et al. (1989) found lower NH_3 volatilization of air-dried poultry manure (6.5 % of applied N) compared to poultry slurry (45.5 % of applied N). The air-dried poultry manure contained 30 % NH_4^+-N of total N compared to 55 % NH_4^+-N of total N for the poultry slurry (Lockyer et al., 1989). Moreover, Lockyer et al. (1989) found a high initial NH_3 volatilization from poultry, pig, and cattle manure and slurry, with 80 % or more of the NH_3 volatilization occurring within the first 48 hours after application. This rapid initial NH_3 volatilization was not observed for the poultry manure containing BBFs included in this study, which all had a delayed response in NH_3 volatilization (Fig. 1). This might be explained by a lower average concentration of 15 % NH_4^+-N of total N in the poultry manure containing BBFs compared to the air-dried poultry manure in Lockyer et al. (1989). Marshall et al. (1998) conducted a study using broiler litter with similar average NH_4^+-N of total N, 14 %, as in our study. Comparable to our observation, Marshall et al. (1998) observed a delayed response in NH_3 volatilization with a sharp increase in the NH_3 volatilization rate 1-3 days after broiler litter application in a field study, using the micrometeorological method.

4.1.5 Animal by-product BBFs

This study included nine BBFs containing animal by-products (Table 1). Accumulated NH_3 volatilization on day 27 from these BBFs surface applied to sand ranged from 6.1 % - 34.3 % of applied N and was generally in the upper range of NH_3 volatilization potential from all BBFs (see Fig. A4). However, the animal by-products containing BBFs was the group with the lowest NH_4^+-N content with 2 % NH_4^+-N of total N. Thus, the relatively high accumulated NH_3 volatilization could not be explained by a high NH_4^+-N content. Therefore, a rapid mineralization of N and subsequent volatilization of the NH_4^+-N likely explains the relatively high NH_3 volatilization. Erwiha et al. (2020) conducted a field experiment on a fine clay loam soil using a semi-static chamber and found NH_3 volatilization of 25 % and 32.1 % of applied N for blood meal and feather meal respectively. The NH_3 volatilization potential from animal by-product derived BBFs in our study is thus comparable, although lower than that found by Erwiha et al. (2020) especially considering that BBFs were applied to sand in our study and to a fine clay loam in the study conducted by Erwiha et al. (2020).

4.2 Experiment 2: Implication of BBF application technique and rate

4.2.1 Application technique

The incorporation of all BBFs reduced potential NH_3 volatilization significantly, except for BO1 (Fig. 2). This is in agreement with numerous previous studies conducted on other BBFs in both field and laboratory trials (Svensson, 1994; Nyord et al., 2012; Feilberg and Sommer, 2013; Herr et al., 2019; Liao et al., 2019; Rocha et al., 2019; Maris et al., 2020).

For the digestate HDG, which was the only BBF with a substantial NH_3 volatilization potential during the first day that was tested for the effect of incorporation, the incorporation reduced the potential NH_3 volatilization markedly by 88 % during the first day. Thus, incorporation was an effective way of preventing initial NH_3 volatilization. Looking at the accumulated NH_3 volatilization for the 27-day period for HDG, it was overall reduced by 64 % though, showing that some NH_3 still volatilized. However, under field conditions where crops will take up some of the NH_4^+, this later NH_3 volatilization might not be as severe as in this laboratory incubation where conditions favoring NH_3 volatilization were applied. From our results, we conclude that incorporation is an effective way to reduce NH_3 volatilization from BBFs, as is also often assumed for more commonly studied BBFs, e.g. manure. Thus, this confirms our hypothesis that incorporation reduces the potential NH_3 volatilization from BBFs.

4.2.2 Application rate

Although a tendency towards a non-linear increase in potential NH_3 volatilization was visually observed for AV4, HDG, and the NH_4HCO_3 reference, it was not clearly significant. This is in agreement with results from a study conducted by Erwiha et al. (2020), who found no clear response of NH_3 volatilization to the BBF application rate of blood meal, feather meal, fish emulsion, and cyano-fertilizer applied on a fine clay loam in a field trial in Colorado, USA. This suggests that the overall trends found in our study are valid over a wider range of application rates than those tested here.

4.3 Experiment 3: Effect of soil type and soil moisture

4.3.1 Soil properties

Soil characteristics and their interaction with time explained 88 % of the variation in accumulated potential NH_3 volatilization from BBFs applied to different soils. Potential NH_3 volatilization was found to negatively correlate with soil pH_{CaCl2} and soil OM. Moreover, a very weak negative correlation was found between potential NH_3 volatilization and soil clay content. The negative correlation observed between soil pH and potential NH_3 volatilization is the opposite of what we expected. This is likely attributed to the fact that the soils expected to have a high NH_3 volatilization due to their alkaline pH also had a high content of clay, which sorbs NH_4^+. Even though Alkaline clay soil had the highest pH, Acidic clay soil and Alkaline clay soil overall had the lowest NH_3 volatilization potentials. Alkaline clay soil had, though, by far the highest CEC and clay content, which must have counteracted the effect of the high pH on the potential NH_3 volatilization. However, the hypothesis that high OM and high clay content decrease the potential NH_3 volatilization can be confirmed.

Our results indicate that it is important to consider the potential interactions of soil characteristics that impact NH_3 volatilization when assessing the impact of the soil on potential NH_3 volatilization. This viewpoint is in agreement with Zhenghu and Honglang (2000), who stress that no single soil property alone can explain NH_3 volatilization. However, it should be emphasized that only five soils were included in this study. Thus, results on the impact of the soil type are not comprehensive, as only a small fraction of variation in soil types was considered here. Despite the incomplete set of soil types included in this study, the effect of the soil type was consistent between BBFs, with the same soils resulting in the highest and lowest NH_3 volatilization potentials, although the magnitude of the difference between soils differed between the different BBFs.

4.3.2 Soil moisture

Mkhabela et al. (2006) found that NH_3 volatilization increased with increasing water filled pore space (WFPS) (50, 70, and 90 % WFPS) from hog slurry applied to a fine loam soil in a laboratory study. However, this was not the case for all BBFs in this study. Only AV4 and partly HDG showed a tendency to increase NH_3 volatilization with increasing soil moisture level. Our finding that there is no clear relationship between NH_3 volatilization potential and soil moisture level is, however, in agreement with other previous studies conducted by Akiyama et al. (2004) and Liao et al. (2019). Liao et al. (2019) found soil moisture to have relatively little impact on NH_3 volatilization, using a dynamic flow-through setup. Akiyama et al. (2004) found no significant effect of different WFPS (40, 60, and 80 %) levels on NH_3 volatilization on a sandy loam soil, using a flow-through chamber technique.

5 Conclusions and implications

The 39 novel BBFs surface applied to sand varied immensely in potential NH_3 volatilization during the 27-day incubation period. CMC and PFC grouping systems for BBFs could not adequately explain the differences in potential NH_3 volatilization from BBFs. However, a grouping of BBFs based on more traditional concepts was markedly better in explaining differences in potential NH_3 volatilization. The biobased fertilizers with the highest accumulated NH_3 volatilization potential were digestates (48 % of applied N volatilized) and BBFs with the lowest accumulated NH_3 volatilization potential were composts and struvites (<1 % of applied N volatilized). Poultry manure, animal by-products, plant-based, and mixed source BBFs had intermediate accumulated NH_3 volatilization potentials in a similar range (12 % to 20 % of applied N). Moreover, the temporal pattern of potential NH_3 volatilization varied greatly between BBFs, with most BBFs showing a delayed response in NH_3 volatilization. The initial BBF characteristics measured in this study and their interaction with time explained 89 % of the variation in accumulated potential NH_3 volatilization from the 39 novel BBFs surface applied to sand.

The temporal patterns of NH_3 volatilization potential suggest that BBF N mineralization dynamics may be a controlling factor. However, this was not measured in the present study. The incorporation of BBFs in soil can effectively reduce potential NH_3 volatilization. However, when incorporation is not an option, e.g. when applying BBFs in growing crops, NH_3 volatilization may pose a significant risk of yield loss and environmental impact for certain BBFs.

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Declaration of competing interest

We declare that we have no known competing interests, neither financially nor personally.

Data availability

All data is openly available online in ERDA, the repository of the University of Copenhagen: https://doi.org/10.17894/ucph.65b73d07-af5f-42f3-9d9e-d6aeab183224

Appendix A

Supplementary data referred to is available in Appendix A.

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