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Agronomic and environmental performance of animal manure-derived ammonium salts vs synthetic mineral fertilisers: 4-year field trial evidence

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ABSTRACT

The European Union (EU) increasing focus on sustainable practices, especially in agriculture, highlights the critical importance of addressing concerns related to the geographical polarity of nutrients and the limited availability of natural reserves. This growing awareness is fueling the adoption of eco-friendly bio-based fertilisers (BBFs) over their mineral counterparts. This study addresses this knowledge gap by subjecting two BBFs, ammonium sulphate (AS) and ammonium nitrate (AN), to a comprehensive 4-year field trial involving crop rotation with maize, spinach, and potatoes at incremental application rates of 40 %, 70 % and 100 % crop total nitrogen (N) demand. The study also evaluates the environmental impacts of these BBFs by comparing gaseous emissions and nitrate leaching risks against their mineral counterparts. Despite challenges arising from the variability in weather conditions during the 4 years of the trial, the selected BBFs demonstrated comparable performance to synthetic ammonium nitrate. The comparative yield range ratio (Yield_{BBF}: Yield_{mineral fertiliser}) ranged from 0.86 to 1.09 for AS and from 0.49 to 1.02 for AN throughout the 4-year field trial duration at 100 %N rate. Moreover, laboratory-based experiments showed significantly lower gaseous emissions for AS (10.39 CO₂eq) and AN (2.81 CO₂-eq) than for their mineral counterpart (15.25 CO₂-eq), likely reflecting slower mineralisation, reduced N₂O emission peaks, and soil pH dynamics relative to the calcium ammonium nitrate reference. Additionally, residual nitrate during winter period from field in the soil remained similar in the case of BBFs and mineral counterparts at all dosages for all 4 years. This indicates that crop uptake and seasonal variability outweighed differences in fertiliser source. These findings emphasize the potential of BBFs to perform at par with mineral fertilisers while offering environmental benefits, making them well suited as a future alternative in circular agriculture.

1. Introduction

Global agriculture stands at the intersection of nutrient distribution, where certain regions are endowed with abundant nutrient reserves, whereas others face a heavy dependence on imports (IPCC, 2023). For example, nearly 70 % of phosphate rock deposits are concentrated in countries like Morocco, China, and the United States (Ryszko et al., 2023), and 80 % of potassium reserves are located in Canada and Russia (Canada, 2024). On the other hand, many European Union (EU) countries are heavily depend on imports to meet their nutrient requirements

for agriculture. In 2022, the EU imported 30 %, 68 % and 85 % of its consumption of inorganic nitrogen (N), phosphates and potash nutrients respectively (Nutrients, 2024).

Geographical nutrient polarity is closely tied to food security. Countries without stable, affordable access to essential nutrients often struggle to maintain high agricultural productivity, impacting food availability and prices (EC, 2024). Their reliance on external sources makes them vulnerable to global price fluctuations, geopolitical tensions, and supply chain disruptions (Zhang et al., 2023; Rethinking agriculture, 2022).

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While certain nutrients like N, can be produced through the energy-intensive Haber-Bosch process, the extraction, processing, and transportation of other nutrients present notable environmental implications (UNEP - UN Environment Programme, 2023). Countries engaged in large-scale nutrient extraction may encounter environmental challenges, including habitat disruption and water pollution (Zahoor and Mushtaq, 2023). The transportation of nutrients over long distances contributes to carbon emissions and energy consumption, further underlining the environmental impact associated with nutrient sourcing and distribution (Juncal et. al., 2023).

Addressing the challenges linked to geographical nutrient polarity requires the implementation of sustainable nutrient management strategies, emphasising the recycling and efficient utilisation of nutrients (European Commission, 2023). A practical solution to mitigate nutrient-related issues involves the implementation of manure-derived bio-based fertilisers (BBFs) as a part of promoting circular agriculture (Hendriks et. al., 2021). To advance this objective, the European Commission (EC) has introduced the concept of RENURE (REcovered Nitrogen from manURE) criteria (Huygens et al., 2020) which, if implemented, would allow to go beyond the current application limit of 170 kg total N/ha/vr set by the Nitrates Directive. In alignment with this initiative, the EC has enacted the Fertilising Products Regulation (Huygens et al., 2019 - FPR, 2019/1009) where manure-derived products must adhere to maximum limit values stipulated in the FPR to be designated as safe fertilising products that can be traded on the EU level. However, the implementation of these regulations has only recently commenced, and there is still a lack of understanding among stakeholders and circular technology developers regarding the newly enforced EU FPR and proposed RENURE criteria (Shrivastava et al., 2024). Consequently, the full potential of BBFs derived from animal manure remains largely untapped.

Several technologies, such as anaerobic digestion, composting, pyrolysis, reverse osmosis, and freeze concentration, are available for the production of BBFs (Dadrasnia et al., 2021). However, among these, stripping-scrubbing has demonstrated the highest efficiency for recovering N as ammonium-based salts (Abbaspour et al., 2024; Brienza et al., 2023). This process entails the conversion of aqueous ammonium (NH_4^+) to gaseous NH3 through pH and temperature adjustments, resulting in the production of ammonium sulphate (AS) or ammonium nitrate (AN) based on the type of acid used in the process (H2SO4 or HNO3) (Brienza et al., 2023; Sigurnjak et al., 2019). Recent studies highlight that recovered ammonium salts consistently deliver agronomic performance comparable to mineral fertilisers, with yields reported at 95-105 % of conventional benchmarks owing to their high solubility and immediate nitrogen availability (Shrivastava and Laasri, 2025). Nevertheless, the majority of this evidence has been generated from greenhouse and pot experiments, while systematic field-scale validation, especially on environmental performance, remains scarce. While AS and AN as BBFs offer several advantages, including precision application, rapid action, and cost-effectiveness (especially in light of the mineral fertiliser price surge triggered by the Russia-Ukraine conflict), past studies have not sufficiently addressed challenges related to gaseous emissions, nitrate leaching, and soil acidification.

Previous research has primarily focused on controlled pot and greenhouse experiments assessing the agronomic performance of AS and AN as bio-based fertilisers (Rodrigues et al., 2022; Saju et al., 2022; Sigurnjak et al., 2019). However, evidence from field-scale trials remains scarce, particularly with respect to long-term crop performance and nitrate leaching. Moreover, information on the environmental behaviour of AS and AN is largely lacking, with little to no data available on their gaseous emission profiles. To address these knowledge gaps, a four-year field trial was conducted to evaluate agronomic performance (crop yield, nitrogen uptake, and nitrogen fertiliser replacement value, NFRV) and nitrate leaching potential, complemented by a short-term laboratory incubation experiment designed to quantify gaseous emissions under controlled conditions. It was hypothesised that (i) BBFs

would sustain crop yields and N uptake comparable to mineral fertilisers, (ii) BBFs would result in lower gaseous emissions due to differences in mineralisation dynamics and soil pH, and (iii) residual post-harvest soil nitrate would remain similar across fertiliser types when applied at equivalent N rates.

2. Materials and methods

2.1. BBFs collection and storage

The AS was obtained from a pig farm equipped with an air scrubbing unit located in Houthulst, Belgium. The AN and pig slurry (PS) were collected from a pig farm in Gistel, Belgium. The pig farm in Gistel has a capacity of 11,000 fattening pigs and can treat 60,000 tonnes of manure each year. For the production of BBFs, centrifugation was used to separate the digestate into solid fraction (SF) and liquid fraction (LF), and the LF was then subjected to NH₃ stripping and scrubbing to extract N. The process is done by capturing the volatile NH₃ in its gaseous form by an acid scrubber, where it is blown through the curtain of dilute nitric acid or sulphuric acid (depending upon the final product required AN or AS) to transfer the ammonia vapours in liquid. The produced AN and AS were stored in the onsite airtight plastic containers (Volume - 200 L -500 L). Airtight polyethylene sampling bottles of 1 L each were used to collect all the BBFs for product characterisation (Table 1). According to the producer's instructions, AN and AS were refrigerated at 4°C. The mineral fertiliser calcium ammonium nitrate (CAN 16 %) was collected from Anorel, Hove (Belgium), and stored according to the producer's instructions.

Dry weight (DW) and moisture content were determined by ovendrying samples at 105 °C for 24 h. The total carbon (TC) and total N (TN) were analysed using a CN analyser (Primacs100 Skalar, Breda, North Brabant, the Netherlands). For ammonium (NHa*–N) and nitrate (NO3 $^-$ –N) analysis, fresh samples were extracted in 200 mL of 1 M KCl and analysed using a San+ + Continuous Flow Analyser (Skalar Analytical BV, Breda, the Netherlands). Macronutrients were analysed by using acid digestion on BBFs samples (1 g sample in 10 mL aqua regia), followed by analysis of extracts via Inductively Coupled Plasma-Optical Emission Spectroscopy (ICP-OES) (Vista MPX, Varian, Inc., Palo Alto, CA, USA). The pH and electrical conductivity (EC) were determined on the freshly obtained sample using an Orion-520A pH meter (USA) and Orion-star A212 conductivity meter, respectively.

2.2. Field trials

The BBFs (AS and AN) were tested in a full-scale field trial in Wingene, Belgium, in a region with predominantly sandy soil texture (Table 2). To assess the agronomic performance of the BBFs at different

Table 1 Chemical composition of tested bio-based fertilisers (AS, AN), pig slurry (PS) and calcium ammonium nitrate (CAN).

Parameters	Unit	AS	AN	PS	CAN
Dry matter	%	21	24	9.60	100
C tot	%	0.07	0.03	3.92	n.d.
N tot	%	4.28	8.75	0.74	16
NH ₄ -N	%	4.28	4.25	0.40	1
NO ₃ -N	%	< 0.01	4.50	< 0.01	15
S tot	%	6.62	n.d.	0.84	n.d.
pН	-	5.24	5.17	7.33	n.d.
EC	mS	199	303	42	n.d.
Ca	%	0.36	0.03	3.53	27
Mg	%	n.d.	n.d.	2.23	n.d.
Na	%	n.d.	n.d.	1.77	n.d.
K	%	n.d.	n.d.	5.70	n.d.
P	%	0.01	0.01	5.01	n.d.

n.d.: not determined; AS: ammonium sulphate; AN: ammonium nitrate; PS: pig slurry; CAN: calcium ammonium nitrate; EC: electrical conductivity

Table 2Chemical characterization of the topsoil layer (0–30 cm) prior to the fertilisation

General soil characteristics	Field Trial	Gaseous emissions
Texture	Sand	Sand
pH (KCl)	5.77 ± 0.20	5.86 ± 0.31
EC (μS/cm)	55 ± 11	59.62 ± 8.57
TN (g/kg)	0.73 ± 0.11	0.77 ± 0.16
NH ₄ *–N (mg/kg)	9.2 ± 0.28	10.1 ± 0.34
NO ₃ -N (mg/kg)	13.1 ± 0.19	11.2 ± 0.15
TC (%)	0.90 ± 0.22	0.97 ± 0.21
Macro-nutrients		
Total P (g/kg)	1.14 ± 0.19	1.08 ± 0.07
Total K (g/kg)	1.02 ± 0.11	1.12 ± 0.04
Total S (g/kg)	0.17 ± 0.02	0.23 ± 0.11
Total Ca (g/kg)	1.26 ± 0.34	1.18 ± 0.26
Total Mg (g/kg)	0.66 ± 0.07	0.75 ± 0.13
Total Na (g/kg)	0.07 ± 0.02	0.09 ± 0.04
Micro-nutrients		
Fe (mg/kg)	4811 ± 654	n.d.
Al (mg/kg)	3588 ± 390	n.d.
Mn (mg/kg)	176 ± 53	n.d.
Zn (mg/kg)	114 ± 56	n.d.
Cu (mg/kg)	49 ± 14	n.d.
Cd (mg/kg)	6.12 ± 4.42	n.d.
Co (mg/kg)	14.60 ± 7.74	n.d.
Cr (mg/kg)	21 ± 11	n.d.
Ni (mg/kg)	38 ± 26	n.d.
Pb (mg/kg)	29 ± 19	n.d.

treatment dosages, a four-year field trial was conducted from 2019 to 2022 in the following crop rotation setting: maize (2019), spinach (2020), potato (2021), and maize (2022). The activity log of 4-year field trial is reported in Annex, Table 1, and visual representation of the field can be found in Saju et al. (2023) where field trial setup and results of the first year are reported. Across 4 years, the following treatments were taken into account:

- Negative control: No fertiliser was added to the plots.
- <u>PK:</u> Only phosphorus (P) and potassium (K) were added to the plots according to the required dosage by the crop; no N was applied.
- <u>NPK:</u> Commercial mineral fertiliser treatment where mineral N (ammonium nitrate 30 %, granulated, broadcasted) was used alongside the required P and K for the crop.
- <u>PS:</u> Plots fertilised with pig slurry. Pig slurry was applied based on the total N dosage, and the required P and K were balanced by using commercial mineral P and K fertilisers.
- AS and AN: Plots fertilised with ammonium sulphate and ammonium nitrate. Ammonium sulphate and ammonium nitrate were applied based on the total N dosage. In this case, P and K were supplied using commercial mineral P and K fertilisers.

All treatments, except for the negative control and PK, received N doses at 40 %, 70 %, and 100 % of the required total N/ha (150 kg N/ha for silage maize, 210 kg N/ha for spinach and 140 kg N/ha for potatoes) as required by the respective crops grown each year (Annex, Table 3). The BBFs were evaluated in comparison to commercial mineral N fertiliser and raw pig slurry. There were four replicates of each treatment (except for negative control and PK, which had eight replicates), resulting in a total of 64 plots. The BBFs were applied using a machine especially designed for field experiments that is equipped with both a vacuum pump and injector coulters for applying organic liquid fertilisers with high viscosity, and a hose pump with a tube system for applying mineral BBFs. This method is used to minimise ammonia volatilisation into the atmosphere. Regarding the supply of macro- and micronutrients, the soil already contained sufficient amounts (except S - details in Annex), so no additional fertilisation was done for these nutrients.

All plots have georeferenced corners. The BBFs at the respective

doses were administered at the same spot on the field for four years. The field was divided into sub-sectors before the start of the trial, and the topsoil layer (0–30 cm) was sampled and studied for soil characterisation (Table 2). Heavy metals, along with macro-/micronutrients, TN, TC, pH (1:2.5 – soil to KCl ratio w/v) and EC (1:5 – soil to water ratio w/v) were analysed using similar methods as mentioned in Section 2.1.

(i) Crops and soil analysis after harvest

At harvest, the crops were severed above (spinach, maize, potatoes leaves) or below (potatoes) the soil surface. Following harvest, the plants were washed to remove any remaining soil (for spinach and potatoes), after which the fresh weight (FW) of yield was determined. Subsequently, the plant samples were subjected to oven drying at $40^{\circ}\mathrm{C}$ for 72 h to determine the dry matter (DM) content. The resultant dry biomass was then pulverized using an industrial grinder, resulting in the ground samples that served as the basis for all subsequent laboratory analysis.

Total N analysis was carried out using a CN analyzer (Primacs100 Skalar, Breda, North Brabant, the Netherlands). Macro- and micro-nutrients such as P, K, and sodium (Na) were investigated through microwave digestion (CEM MARS 5, Drogenbos, Flemish Brabant, Belgium) of plant samples (0.2 g sample in 10 mL HNO₃ (65 %)). The digested solutions were subsequently analyzed using Inductively Coupled Plasma-Optical Emission Spectroscopy (ICP-OES) (Vista MPX, Varian, Inc., Palo Alto, CA, USA).

For soil analysis, using an auger, a representative soil sample was taken from each plot using a 5-point sampling approach (the centre and the four corners) (Hendriks et al., 2021) at three different soil depths (0-30 cm, 30-60 cm, and 60-90 cm). Fresh soil samples were placed in a plastic bag and transferred to a lab. The first step was to clean soil from potential presence of roots, after which soil was thoroughly mixed to obtain a homogenous sample per plot and soil layer. The fresh soil samples, from all three soil layers, were used to determine the moisture content and mineral N content. The soil moisture content was assessed by drying a portion of the fresh soil sample at 105°C for 24 h. Other portion of fresh soil was used to determine mineral N content by digesting fresh soil in 200 mL 1 M KCl to determine contents of NH₄-N and NO₃—N determined on a San⁺⁺ Continuous Flow Analyser (Skalar Analytical BV, Breda, the Netherlands). The remaining fresh soil underwent air-drying and sieving using a 1 mm sieve. Air-dried soil samples, from 0 to 30 cm soil layer, were characterised for TC, TN, pH, and EC using the same protocols outlined in start of Section 2.1.

(ii) Weather monitoring

During the entire trial duration, weather conditions were closely monitored using a weather station positioned at a nearby farm, situated within a distance of less than 500 m. The measurement of precipitation levels employed the tipping bucket method, while hourly temperature readings were recorded (Fig. 1). The weather monitoring provides crucial data for understanding and interpreting the trial results in the context of varying climatic conditions.

2.3. GHG emissions

The emissions of CO_2 and N_2O were measured over an 18-day incubation period using a Gasera One Multi-gas analyser (Turku, Finland) equipped with a photo-acoustic infrared analyser. The soil mesocosms used for the incubation tests were 1 L Duran bottles modified with GL45 - thread Smart caps (model: SW45–2A). The experiment consisted of 6 products – one negative control, two mineral fertilisers (46 % urea and 16 % CAN as positive controls), raw pig manure and two BBFs (AN and AS). Following the Nitrates Directive, all fertilisers were mixed properly and administered to 568 g of pre-incubated soil at a rate of 170 kg total N/ha (Table 2). Each mesocosm was maintained at 80 % water-filled

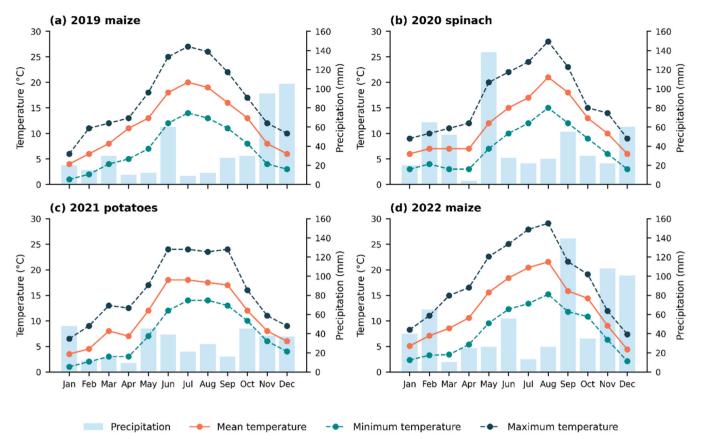


Fig. 1. Daily precipitation and average temperature conditions during the trial period in 2019 - 2022 field trials (partially retrieved from Saju et al., 2023).

pore space (WFPS) throughout the experiment, with moisture levels adjusted by periodic weighing and addition of deionised water. A WFPS of 80 % was selected as it is commonly applied in sandy soil incubation studies to stimulate denitrification and capture $N_{\text{2}}O$ emission dynamics (Egene et al., 2022; Shrivastava et al., 2023). Using the change in concentration over time and accounting for the volume of the headspace, the tubing, and the area of the soil surface, fluxes of CO_2 and N_2O were calculated.

On days 0, 1, 2, 4, 7, 9, 11, 14, 16, and 18, the measurement was done. Two 1 m long Teflon tubes with a 2 mm inner diameter were used to create a closed circuit to connect the analyser to the mesocosm. Gases were removed from the headspace (at a flow rate of 800 mL/min), processed by the analyser, and then reintroduced to the mesocosm in a closed loop during the measurement. After attaching the tube to the mesocosm, gas concentrations in the headspace were monitored after 4, 8, 12, and 16 min. The analyser picked up the change in gas concentration during each 4-minute interval. The fluxes of CO_2 and CO_2 and CO_3 for the gaseous emissions were computed using concentration changes over time, accounting the headspace capacity, the pipework, and the size of the soil surface. Using the ideal gas law, gas concentrations (measured in ppm) were converted to emission flux:

$$Flux_{area} = \frac{\Delta gas}{\Delta t} \times \frac{P \times M \times n}{R \times T} \times \frac{V}{A}$$
 (3)

Where, flux area is the elemental flux released as a gas, in $\mu g \ m^{-2} \ h^{-1}$ or $\mu g \ kg^{-1} \ h^{-1}$; $\Delta gas/\Delta t$ is the slope of the linear regression of gas concentration against time; P is the pressure in the cell (0.838 atm); M is the molar mass of the element (e.g., 14 for N); n is the number of atoms of the element in the gas (e.g., 2 N in N₂O); R is the ideal gas constant (0.08206 L atm $mol^{-1} \ K^{-1}$); T is the average atmospheric temperature (294 K); V is the total volume of the headspace, tubing, and analyser cell (0.623 L); and A is the surface area of the soil in the mesocosm

 $(0.0069 \text{ m}^2).$

The cumulative flow for each gas was calculated using linear interpolation between two measurement days. The cumulative fluxes obtained with the soil control were subtracted from all cumulative emissions of fertiliser.

2.4. Data and statistical analysis

The data were analysed using a one-way ANOVA (p < 0.05) to determine significant differences between treatments. Additionally, a two-way ANOVA (p < 0.05) was conducted to evaluate the interaction effects between treatment and dosage, with treatment and dosage defined as fixed factors. The response variables analysed as dependent variables included fresh yield, dry yield, nitrogen uptake (N-uptake), nitrogen fertiliser replacement value (NFRV), and fertiliser replacement use efficiency (FRUE). The univariate general linear model in SPSS 26 was utilised for these analyses. Tukey's Honest Significant Difference (HSD) test was applied for post-hoc comparisons. In tables and figures, uppercase letters (A - D) denote statistically significant differences in the means among different N dosages (e.g. AS 40 % vs. AS 70 % and AS 100 %) for each year, while lowercase letters (a, b, c, d) denote significant differences among different treatments for each year (e.g. AS 40 % vs. AN 40 % vs. NPK 40 %). If no letters appear within a table or figure, this indicates an absence of statistically significant differences. For clarity and uniformity, nonsignificant comparisons have been omitted. The figures were generated in Python using JupyterLab, based on the processed experimental datasets and standardized plotting scripts.

Based on the physiochemical data from the pot trials, apparent N recovery (ANR) and N fertiliser replacement value (NFRV) were assessed. ANR and NFRV are calculated in the following equations (Cavalli et al., 2016; Schröder et al., 2014; Sørensen and Eriksen, 2009):

$$ANR_{fertiliser} = \left(\frac{crop\ N\ uptake_{fertiliser} - crop\ N\ uptake_{control}}{total\ N\ applied_{fertiliser}}\right) \tag{4}$$

$$NFRV(\%) = \frac{ANR_{BBF}}{ANR_{CAN}}$$
 (5)

Furthermore, the calculation of fertiliser use efficiency (FUE) and fertiliser replacement use efficiency (FRUE) were conducted for BBFs and PS. The FUE and FRUE were calculated similarly to ANR and NFRV, however, the effect of N uptake from unfertilised treatment is not considered. This was majorly done to avoid the high standard deviations caused due to the varying effect of unfertilised treatment; especially in N-rich soils. The FUE and FRUE calculations were done as follows (Sigurnjak, 2017):

$$FUE(kg\ ha^{-1}) = \frac{N\ uptake_{treatment}(kg\ ha^{-1})}{Total\ N\ applied_{Treatment}(kg\ ha^{-1})} \tag{6}$$

$$FRUE(\%) = \frac{FUE_{BBF}}{FUE_{Mineral N fertiliser}}$$
(7)

3. Results

3.1. Agronomic performance

3.1.1. Fresh yield and dry yield in field trials

Across the 4-year field trial, notable differences among treatments and N dosages for yield were observed. In the first year of silage maize trial, no significant differences were observed among the treatments. However, in the second year with spinach, significant differences were noted, with AS and PS performing significantly better than other treatments (Table 3). In the third year with early potatoes, while no significant differences were observed among BBFs and synthetic fertilisers, all treatments outperformed the negative control (Table 3). In the fourth year with silage maize, AS and AN showed the highest performance in some cases, significantly surpassing other treatments at varied N rates across the trial.

Regarding dosages, the first year with silage maize saw no significant differences among the dosages. In contrast, the second year with spinach revealed significant differences, with 100 % dosage performing significantly better than 40 %, and notable differences observed between 70 % and 100 % (Table 3). In the third year with early potatoes, dosages of 70 % and 100 % performed equally well, both significantly outperforming 40 % dosage (Table 3). Finally, in the fourth year with silage maize, no significant differences were found among the dosages of 70 %, 40 %, and 100 %, however all dosages performed better than the negative control (Table 3).

3.1.2. N uptake and replacement potential

In 4-year field trial, the two-way ANOVA analysis revealed notable differences among treatments and dosages for N uptake. For treatments, in the first year with silage maize, a similar N uptake was observed with synthetic fertiliser as compared to BBFs (p >0.05). In the second year with spinach, N uptake was significantly higher (p ≤ 0.05) for AN and NPK compared to negative control and PK (Fig. 2). During the third year with early potatoes, all treatments performed similar, though every treatment had significantly higher N uptake compared to the negative control (p ≤ 0.05). In the fourth year with silage maize, the synthetic fertiliser again showed the on average higher N uptake (p >0.05).

Two-way ANOVA for dosages (Table 4), showed the first year with silage maize the 40 % and 100 % N dosage resulted in the significantly (p ≤ 0.05) higher uptake compared to negative control. In the second year with spinach, both 70 % and 100 % N dosages performed on average higher (p >0.05) than the 40 % dosage, with 100 % showing the highest uptake. In the third year with early potatoes, dosages of 70 % and 100 % had similar N uptake, both on average higher than the 40 % dosage (p >0.05). In the fourth year with silage maize, the 40 % and 100 % N dosages showed the highest uptake, significantly higher than the negative control dosages.

In the context of N fertiliser replacement potential for 2019 silage maize, no significant differences between treatments were observed for ANR (Annex, Table 5) and NFRV (Table 5). Notably, the treatment involving 70 % N dosage of AN exhibited the highest NFRV (148.99

Table 3 Mean fresh matter yields and dry matter yield (crops harvested in t/ha) for treatments at different dosages in 2019 – 2022 field trials. Negative control and PK are used as standards and are similar for all dosages within a particular year. Replicates n=8 for negative control and PK, n=4 for all treatments.

Product	2019 (Silage maize)		2020 (Spinach)		2021 (Early potatoes)		2022 (Silage maize)	
	Fresh matter yield	Dry matter yield	Fresh matter yield	Dry matter yield	Fresh matter yield	Dry matter yield	Fresh matter yield	Dry matter yield
Negative Control	31.6 ± 6.8	13.3 ± 2.4	A 2.8 \pm 2.6 a	A 0.4 ± 0.4 a	A 12.0 \pm 1.8 a	A 2.8 \pm 0.4 a	A 24.9 \pm 4.8 a	A 7.2 \pm 1.7 a
PK	30.1 ± 5.9	12.2 ± 2.1	A 2.3 \pm 1.8 ab	A 0.3 ± 0.2 ab	A 12.4 \pm 3.2 a	A 2.9 \pm 0.6 a	A 25.7 \pm 4.2 ab	A 6.6 \pm 1.4 ab
NPK 40	34.8 ± 6.8	12.0 ± 3.7	AB 4.1 \pm 2.6 ab	AB 0.4 ± 0.2 ab	A 17.9 \pm 6.0 b	A 4.1 \pm 1.3 b	B 29.6 \pm 5.7 bc	B 7.6 \pm 1.2 bc
Pig slurry 40	32.0 ± 3.7	12.2 ± 1.0	AB 10.2 \pm 6.7 b	AB 1.1 \pm 0.7 b	A 15.2 \pm 4.9 b	A 3.3 \pm 0.3 b	B 26.5 ± 2.3 abc	B 7.1 ± 0.5 abc
Ammonium nitrate (AN) 40	33.0 ± 9.1	12.8 ± 2.3	AB 3.4 \pm 1.5 ab	AB 0.4 \pm 0.2 ab	A 15.3 \pm 4.9 b	A 3.3 \pm 1.0 b	$\text{B 34.3} \pm 1.8\text{c}$	$B~9.1\pm0.6c$
Ammonium sulphate (AS) 40	31.8 ± 6.5	11.5 ± 3.0	AB 13.2 \pm 13.2b	AB 1.6 \pm 1.4 b	A 16.6 \pm 5.5 b	A 3.6 \pm 1.1 b	$B~32.2\pm1.9c$	$\text{B 7.8} \pm 0.1\text{c}$
NPK 70	32.3 ± 7.6	11.7 ± 3.9	BC 14.8 \pm 9.4 ab	BC 1.9 ± 1.2 ab	B 22.1 \pm 6.7 b	B 4.9 \pm 1.4 b	B 29.2 \pm 5.9 bc	B 7.1 \pm 0.9 bc
Pig slurry 70	31.2 ± 6.2	10.4 ± 1.8	BC 15.8 \pm 9.1 b	BC 2.1 \pm 1.3 b	B 23.1 \pm 2.7 b	B 4.9 \pm 0.5 b	B 28.2 \pm 3.2 abc	B 6.7 \pm 0.8 abc
Ammonium nitrate (AN) 70	30.3 ± 9.7	11.5 ± 2.4	BC 7.6 \pm 1.3 ab	BC 0.9 \pm 0.1 ab	B 23.2 \pm 2.7 b	B 5.1 \pm 0.6 b	$B~32.1\pm2.8c$	$\text{B 7.6} \pm 1.1\text{c}$
Ammonium sulphate (AS) 70	29.7 ± 2.4	10.6 ± 2.5	BC 11.6 \pm 10.4b	BC 1.2 \pm 0.9 b	$B~25.9\pm6.2~b$	B 5.7 \pm 1.3 b	$B~28.2\pm4.9c$	$B~6.9\pm1.1c$
NPK 100	35.6 ± 7.4	12.7 ± 2.3	C 18.4 \pm 12.5 ab	C 2.0 ± 1.2 ab	B 22.2 \pm 3.9 b	B 5.0 \pm 0.6 b	B 32.9 \pm 5.9 bc	B 8.5 \pm 1.8 bc
Pig slurry 100	26.9 ± 4.7	9.7 ± 2.1	C 16.5 \pm 10.0 b	C 1.9 \pm 1.1 b	B 29.1 \pm 8.1 b	B 6.4 \pm 1.8 b	B 26.9 \pm 1.5 abc	B 7.4 \pm 0.4 abc
Ammonium nitrate (AN) 100	31.8 ± 9.9	10.5 ± 2.9	C 10.2 \pm 4.2 ab	C 1.0 \pm 0.4 ab	B 21.1 \pm 7.4 b	B 4.6 \pm 1.5 b	$B~29.3\pm5.2c$	$\text{B 7.2} \pm 1.4\text{c}$
Ammonium sulphate (AS)	36.5 ± 6.6	12.1 ± 2.1	C 16.2 \pm 14.0 b	C 1.6 \pm 1.3 b	B 24.0 \pm 7.1 b	B 5.3 \pm 1.4 b	B 34.8 ± 2.6c	$B~8.4\pm1.2~c$

(N)PK: synthetic fertiliser of (nitrogen), phosphorous and potassium. Lower case letters (a–d) denote statistically significant differences in means among the treatments for individual year (Tukey's Test for p < 0.05). Uppercase letters (A-D) denote statistically significant differences in means among dosages of N applied for individual year for single treatment (Tukey's Test for p < 0.05). In case of absence of any letters signifies that no differences were seen for that particular year in treatments and dosage.

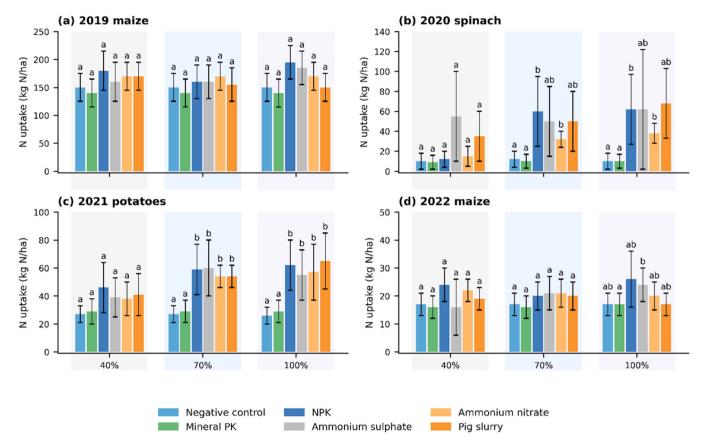


Fig. 2. Mean N uptake by plants for 2019-2022 field trials. Negative control and PK do not contain N, and hence are similar for all dosages. Standard deviation is represented by error bars (n = 8 for negative control and PK, n = 4 for all other treatments). Lower case letters (a–d) denote statistically significant differences in means for one-way ANOVA (Tukey's Test for $p \le 0.05$) among the products and dosages.

 $\label{thm:continuous} \begin{tabular}{lll} \textbf{Table 4} \\ \textbf{Mean yields (with Tukey HSD groupings) of crops across four years under different N dosages. Values in parentheses indicate homogeneous groups determined by Tukey's Honestly Significant Difference (HSD) test at p < 0.05. Treatments sharing the same letter within a year are not significantly different. } \end{tabular}$

Dosage (% N required by crop)	Year 1 Yield (Silage Maize)	Year 2 Yield (Spinach)	Year 3 Yield (Potatoes)	Year 4 Yield (Silage Maize)
0	145.62 (A)	8.60 (A)	28.45 (A)	16.69 (A)
40	171.38 (B)	28.71 (B)	40.78 (A)	21.78 (B)
70	162.75 (AB)	48.34 (C)	56.83 (B)	20.62 (B)
100	176.95 (B)	58.31 (D)	59.88 (B)	21.70 (B)

 \pm 140.79), albeit with a considerable standard deviation, suggesting notable variability among replicates. Conversely, the treatment using AS at 40 % N dosage displayed the lowest NFRV (51.93 \pm 67.86).

3.2. Environmental performance

3.2.1. Nitrate leaching risk

One of the essential aspects of the safe application of BBFs is the to assess the risk for nitrate leaching into the ground and surface water (Fig. 3) by measuring the nitrate residue in the 0–90 cm soil layer in the period October 1st – November 15th. Across the years, the nitrate residue in the soil during the winter period was generally found to be higher than the legal limits imposed by Flemish government. However,

Table 5 NFRV and FRUE (mean \pm standard deviation) values for BBFs in comparison to synthetic N fertiliser at different treatment dosages for 2019–2022 field trials.

	2019 (Silage maize) 150 kg/ha		2020 (Spinach) 207 kg/ha		2021 (Early potatoes) 140 kg/ha		2022 (Silage maize) 150 kg/ha	
TN applied Product								
	NFRV (%)	FRUE (%)	NFRV (%)	FRUE (%)	NFRV (%)	FRUE (%)	NFRV (%)	FRUE (%)
Pig slurry 40	105 ± 59	93 ± 9	552 ± 569	284 ± 230	81 ± 98	88 ± 31	37 ± 63	78 ± 17
Ammonium nitrate (AN) 40	73 ± 66	94 ± 15	146 ± 158	118 ± 62	47 ± 80	80 ± 29	54 ± 30	90 ± 12
Ammonium sulphate (AS) 40	51 ± 67	89 ± 15	853 ± 1046	415 ± 434	51 ± 64	82 ± 23	-8.8 ± 135	65 ± 44
Pig slurry 70	101 ± 220	95 ± 20	77 ± 50	82 ± 46	99 ± 19	92 ± 8.2	135 ± 149	102 ± 21
Ammonium nitrate (AN) 70	148 ± 140	106 ± 18	42 ± 10	49 ± 9	85 ± 23	92 ± 11	113 ± 82	107 ± 17
Ammonium sulphate (AS) 70	90 ± 114	98 ± 15	79 ± 66	86 ± 62	104 ± 71	102 ± 35	129 ± 155	105 ± 26
Pig slurry 100	28 ± 76	77 ± 14	104 ± 73	107 ± 66	120 ± 68	101 ± 31	4.0 ± 48	64 ± 15
Ammonium nitrate (AN) 100	56 ± 74	87 ± 21	55 ± 21	60 ± 19	82 ± 66	90 ± 35	24 ± 31	74 ± 15
Ammonium sulphate (AS) 100	88 ± 68	96 ± 19	94 ± 102	100 ± 95	76 ± 57	87 ± 30	71 ± 40	89 ± 15

TN: total nitrogen; NFRV: nitrogen fertiliser replacement value; FRUE: fertiliser replacement use efficiency; (N)PK: synthetic fertiliser of (nitrogen), phosphorous and potassium. The Tukey HSD letters were not shown in the table as no significant differences were found.

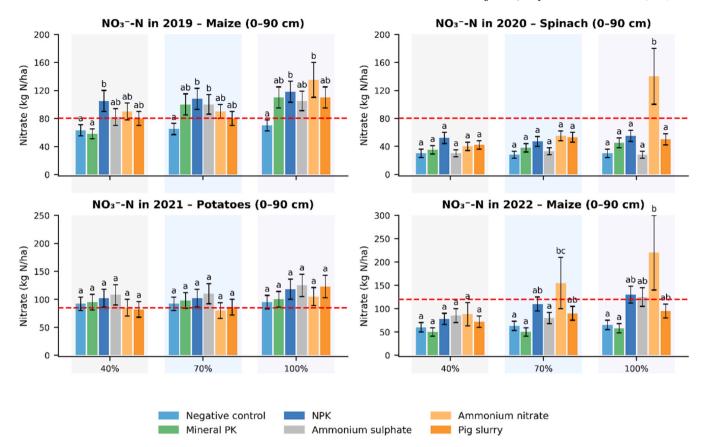


Fig. 3. Mean nitrate-N (NO₃-N) (in kg/ha) - in the soil profile (0–90 cm). Negative control and PK do not contain N, hence are similar for all N dosages in 2019–2022 field trial. Red dotted line denotes Flemish legislative limit in winter period for nitrates. Standard deviation is represented by error bars (n = 8 for negative control and PK, n = 4 for all other treatments). Lower case letters (a–d) denote statistically significant differences in means (Tukey's Test for $p \le 0.05$) among the products and dosages.

no significant difference in the risk of leaching was observed between BBF treatments and their mineral counterpart (details of nitrate residue at different depths in Annex Table 6).

Soil nitrate (NO₃⁻N) concentrations in the 0–90 cm profile varied across years, crops, and treatments. In 2019 (maize), values ranged between 60–120 kg N ha⁻¹, with significantly higher concentrations observed under some NPK and AN treatments at 100 % dosage compared to the control (Fig. 3). In 2020 (spinach), nitrate levels were lower overall (30–80 kg N ha⁻¹), except for PS at 100 % dosage, which showed a marked increase (>150 kg N ha⁻¹). In 2021 (potatoes), nitrate concentrations were more uniform across treatments (80–160 kg N ha⁻¹), with no significant differences among fertilizer types or dosages (Fig. 3). In 2022 (maize), nitrate accumulation was more pronounced, particularly under PS at 100 % application, which exceeded 300 kg N ha⁻¹, while most other treatments remained below 150 kg N ha⁻¹ (Fig. 3).

3.2.2. Gaseous emissions

 $3.2.2.1.\ N_2O$ emission. To complement the field trial, a short-term incubation experiment was conducted under controlled soil moisture conditions. Such incubations allow direct comparison of gaseous emission dynamics between fertiliser treatments, minimising the influence of variable weather conditions. This approach provides mechanistic insights that cannot be easily captured under field conditions alone.

Cumulative N_2O-N emissions showed clear treatment differences over the 18-day incubation period (Fig. 4a). Urea consistently produced the highest emissions, with a sharp increase between days 2 and 7, reaching around 16 mg N_2O-N m⁻², and continuing to rise steadily to approximately 23 mg N_2O-N m⁻² by day 18. CAN also showed a rapid

early increase (Annex Figure 12), reaching about 10 mg N_2O-N m⁻² by day 4 and stabilizing after day 14 at around 14–15 mg N_2O-N m⁻².

PS displayed an early peak of around 8 mg $N_2O-N\ m^{-2}$ on day 4, after which emissions declined slightly and remained nearly stable throughout the rest of the period (Fig. 4a). In contrast, AN and AS exhibited comparatively lower cumulative emissions, showing a more gradual increase across the experiment and ending at about 9 and $12\ mg\ N_2O-N\ m^{-2}$, respectively.

3.2.2.2. CO_2 and CH_4 emissions. Cumulative CO_2 –C emissions varied considerably among treatments over the 18-day incubation. Pig slurry (PS) showed the highest emissions, increasing steadily from about 3 g CO_2 –C m⁻² at day 0 to more than 12 g CO_2 –C m⁻² by day 18 (Fig. 4b). Urea also resulted in elevated emissions, rising gradually to approximately 7 g CO_2 –C m⁻² by day 7 and then stabilizing at this level for the remainder of the experiment. In contrast, CAN displayed a distinct decline, with values dropping continuously after day 2 and reaching around -7 g CO_2 –C m⁻² by day 18. Both AN and AS showed minimal changes, remaining close to baseline with cumulative emissions fluctuating slightly around zero throughout the incubation (Annex Figure 13).

Cumulative CH₄–C fluxes showed consistent net uptake across all treatments during the 18-day incubation. PS and AN exhibited the strongest declines, reaching around -32 mg CH₄–C m⁻² and -27 mg CH₄–C m⁻², respectively, by day 18 (Fig. 4c). AS also showed substantial uptake, decreasing to about -26 mg CH₄–C m⁻². Urea treatments displayed a moderate decline, stabilizing at approximately -20 mg CH₄–C m⁻² after day 14. In contrast, CAN resulted in the smallest reduction, with fluxes decreasing gradually to only about -8 mg CH₄–C m⁻².

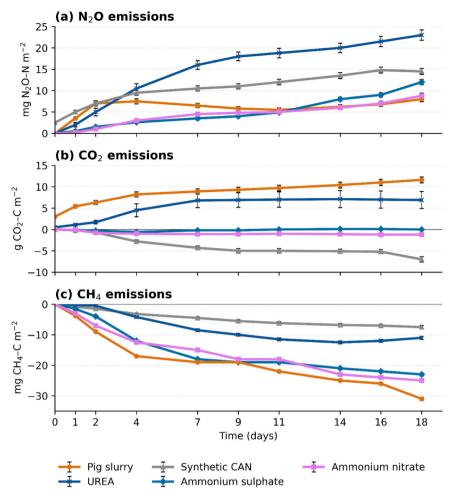


Fig. 4. a) Cumulative emissions of nitrous oxide (mg N_2O -N per m_2 of soil), b) Cumulative emissions of carbon dioxide (g CO_2 -C per m^2 of soil) and c) Cumulative emissions of methane (mg CH_4 -C per m_2 of soil). Legend: AS—ammonium sulphate (AS), UREA N – 46 % UREA, CAN—calcium ammonium nitrate, PS – pig slurry and AN – ammonium nitrate (AN). The values are obtained after subtracting negative control from all the treatments.

4. Discussion

4.1. Agronomic performance

4.1.1. Biomass yield in field trials

In highly fertilised agricultural regions such as Flanders, soils tend to be nutrient-rich, making direct comparison between fertilised and unfertilised treatments challenging. Although there was no significant difference between treatments or dosages in 2019 silage maize trial, it is important to note that this trial resulted in overall lower yields compared to field trials conducted with similar BBFs in previous studies in the similar geographical region. In the study conducted by Sigurnjak et al. (2019), a field trial focusing on silage maize as the test crop revealed that BBFs (AN and AS) exhibited approximately 80 - 90 % higher fresh matter yield, (59 \pm 6 tonnes ha⁻¹ for AN and 59 \pm 4 tonnes ha⁻¹ for AS). Similarly, another study by Vaneeckhaute et al. (2014) demonstrated increased yield results in the range of 100 - 120 % higher fresh matter yield (73-81 tonnes ha-1) for maize. The lower yield observed in this study could be attributed to weather-related effects on the maize growing period. The availability of soil moisture is a critical parameter influencing crop growth, and the temperature and moisture levels during key physiological growing periods significantly impact the final maize yield (Yin et al., 2023). In May 2019, unusually colder and dryer conditions prevailed (Fig. 1), which were suboptimal for maize cultivation. This period of lower temperatures was succeeded by a sudden transition to extremely hot and dry conditions in July, leading to an early conclusion of the vegetative phase in maize development. These climatic variations likely contributed to the observed decrease in yield (Saju et al., 2023). However, the yields from Sigurnjak et al. (2019) and Vaneeckhaute et al. (2014) are towards the higher end of the spectrum in this temperate zone with average yields around 50-60 tonnes ha $^{-1}$.

In 2020, the variability in spinach production was significant due to extremely dry conditions and wind erosion (Fig. 1). For instance, in the case of spinach, generally, 200-250 liters per square meter of water is required for overall growth (Bianchi, Masseroni and Facchi, 2017). However, in our trials, only 20–25 liters were supplied through rainfall, with an additional 50 liters applied via surface irrigation, creating a deficit that reduced spinach yields. This lower water application was primarily due to drought conditions in Belgium, which led to crop failures across the trial region. Additionally, the soil quick-dry nature prevented effective water retention, further contributing to lower yields in spinach. As a result, even if differences were observed, they may not be reliable. In terms of fresh matter yield, both the negative control and PK showed significantly inferior performance compared to all treatments tested at 70 % and 100 % of the N dosage, except for AN (Table 5). Notably, there was no significant difference between 100 % and 70 % of the N dosage for all treatments. This observation might imply that the tested soil conditions already have sufficient N supply. Therefore, any additional input of N, which is typically recommended, could lead to potential environmental losses. Moreover, a similar pattern is observed for dry matter yield (Table 5), where AN at 40 % N dosage performed significantly inferior than all other treatments. Even at 70 % and 100 %

of the N dosage, AN demonstrated a lower yield on average (approximately 50-55 % lower) compared to AS, PS and NPK. Similar yields were observed for AS and synthetic fertiliser NPK across all N dosages. The results aligns with findings from a study by Rodrigues et al. (2022), where waste-recovered AS and AN exhibited comparable performance to commercial synthetic fertilisers for radish and spinach pot cultivations. The performance of AN appears to be influenced by both weather conditions and cultivation practices. The exceptionally dry weather, particularly during the period between sowing and the initial irrigation, heightened the vulnerability of seedlings to drought-related stress (Seleiman et al., 2021). Additionally, the adoption of non-reversing soil tillage practices resulted in the BBFs being retained in the topsoil layer, exerting an immediate impact on the seedlings. It is plausible that the application of AN induced salt-related stress compared to the other treatments (Ramezanifar et. al., 2022). However, an examination of the soil EC after harvest exhibited the lowest results for AN-treated crops. Therefore, this reason may not be applicable in explaining the observed phenomenon.

In 2021, early potatoes were selected as a test crop. Similar to 2020, no significant differences were observed between the 70 % and 100 % N dosages. This is likely due to cultivation in N-rich soil, which further equalises the yield of early potatoes at both 70 % and 100 % N dosages. While N may not have been the sole limiting factor in this trial, the results, along with findings from other regional studies, suggest that additional N application may not be necessary. Other factors, such as soil compaction, texture (particularly sandy soils), and agronomic management practices, could also have played a role in the lower yields observed. Given the historically N-rich nature of soils in this region, optimising N use remains a priority to avoid unnecessary applications. The fresh matter yield trajectory is also followed by the dry matter yield (Table 5). On average, a lower yield observed with AN in comparison to AS and PS, but no significant differences could be observed (p > 0.05). No significant differences were observed in yield between AS and NPK for all N dosages. Similar results were observed by Hendriks et al. (2021), where AS tested on early potatoes resulted in comparable yields to those with synthetic fertiliser. Variability was high during the trial due to the moderately rainy weather (Fig. 1), which made it challenging to find any statistical differences. In comparison to pig slurry at 100 %dosage, both AN and AS exhibit a notably lower performance on average (p > 0.05). This could be majorly attributed to 1) the leaching risk out of AS and AN (100 % in mineral form) at the start of field trial due to heavy rain (Nyamangara et al., 2003) and, 2) the PS contained organic N which did not leach and got mineralised in later stages of crop growth. There is also a significant effect of the N dosage applied (p < 0.05) - in the classes between 35 and 70 mm, the more N supplied, the higher the yield (Annex, Table 2). In smaller and larger classes, no significant differences could be observed. In the size class between 35 and 50 mm, the yield was significantly higher for PS compared to AN, AS, and synthetic NPK. This might be primarily attributed to the high percentage of readily available N in the fertilisers (NPK, AN, and AS), which enhances the immediate availability of nutrients, making them more prone to leaching risk.

In 2022, field trials in Belgium were scheduled from June to October using silage maize as a test crop. However, unforeseen challenges arose, including exceptionally high temperatures in July, reaching around 40C in the fields, followed by drought conditions in August (Fig. 1). The emergency harvest was initiated due to decreasing dry matter content in the silage maize, as drought conditions caused excessive drying. Silage maize requires a moisture content of 60–70 % for optimal wet-packing, and further delays could have resulted in significant yield losses. Harvesting at moisture levels below 60 % can result in poor packing, inadequate air exclusion, and poor fermentation, leading to higher spoilage and reduced silage quality (Bagg et al., 2007). Therefore, delaying harvest beyond the optimal moisture range can cause significant yield losses and compromise silage quality. However, significant differences in both fresh and dry matter yields were observed among fertilisation treatments (p < 0.05). Treatments with AN and AS,

particularly at 40 % and 100 % N rate, consistently produced the highest yields and were significantly superior to the control and PK (p < 0.05). These mineral N sources showed a clear advantage in both fresh and dry matter production, likely due to their readily available N content. In contrast, PS although somewhat improved over the negative control, often showed no statistically significant difference (p > 0.05) and demonstrated more variable performance. NPK treatments provided intermediate results, generally improving yields compared to the control but not reaching the levels achieved by AN and AS (p < 0.05). Overall, the results suggest AN and AS, were more effective in enhancing silage maize productivity under the 2022 conditions (p < 0.05). Similar to the findings from 2019, a marked reduction in maize biomass was observed in 2022 when compared to typical values reported in other studies. Overall, biomass production was reduced by approximately 50-55 %, likely due to adverse climatic conditions. These results are consistent with those of Kamara et al. (2003), who reported a 50 % decline in biomass under drought stress. Additionally, Lobell et al. (2020) observed a sharp decline in maize yield when temperatures exceeded 30°C, suggesting that heat stress may have further contributed to the observed yield reduction in 2022.

4.1.2. N uptake and NFRV

AS and AN exhibited performance similar to synthetic fertiliser with respect to FUE and FRUE across all N dosages. In study by Sigurnjak et al. (2019), the ANR values (0.42–1.07 for AN and 0.55–0.73 for AS) for silage maize treated with AN and AS exceeded the findings obtained in the current experiment (Annex, Table 5). The FUE outcomes (Annex, Table 5) of the treatments support the hypothesis that a substantial portion of N uptake in the crop may originate from native soil N, rather than from the applied fertiliser. The N deficit, coupled with reduced water availability during the trial period, led to crops treated with PS actively scavenging the soil for all available N, resulting in an enhanced FUE compared to other treatments where N was provided in ample quantities.

For 2020 trials with spinach, AN displayed on average a lower N uptake than other treatments at all N dosages (p > 0.05) (Fig. 2). This lower N uptake of AN explains the lower yield as discussed in Section 3.1.1. The ANR and FUE values appear consistently low across all treatments (Annex, Table 5). This could be attributed to the total N uptake in all instances being approximately 100 kg N/ha lower than the supplied N amount. Additionally, the initial nitrate content in the soil before sowing was low, and the dry conditions during the trial further contributed to reduced N-mineralisation (Brackin et al., 2019). While the ANR and FUE values for AS (0.25 \pm 0.27; 0.64 \pm 0.61) and synthetic fertiliser (0.27 \pm 0.19; 0.63 \pm 0.39) were quite similar, it is noteworthy that the ANR of AN (0.15 \pm 0.06; 0.39 \pm 0.12) is notably inferior than all other treatments. A similar trend is observed for NFRV and FRUE (Table 5), where AS and PS exhibited performance comparable to synthetic fertilisers at 100 % of the N dosages. In some instances, NFRV/FRUE values exceeded 100 for certain treatments (for 40 % of N dosage); however, the considerable standard deviation in those cases was attributed to the impact of weather conditions on the overall trial.

For the 2021 field trials with early potatoes, an effect of the recommended dosages on the N content measured in tubers (Fig. 2) is significantly visible across treatments. The N uptake in leaves, tubers, and total N uptake correlates with the total N supply, indicating that lower N dosages result in reduced N uptake. The average N uptake from PS was higher as compared to AS and AN at 100 % of the applied dosage (p > 0.05) (Fig. 2). Similar to the yield, the performance of 70 % of the N dosage is similar to 100 % of the N dosage. NFRV was low in all treatments, possibly caused by the leaching out of significant amounts of the applied N before it could have been taken up by the crop (Table 5). The differences between the 3 dosages applied were also reduced, as more nitrate leached from plots receiving higher N dosages. The NFRV value of PS 100 % was highest compared to BBFs. Additionally, the differences between the 3 dosages are also high where PS was applied. Moreover,

the NFRV value for AS at 70 % is > 100 showing the potential of replaceability of conventional synthetic fertiliser with the AS. Identical results were also observed by Hendriks et al. (2021), where an NFRV of 113 % for AS in early potatoes was observed, demonstrating its performance comparable to synthetic fertilisers. Similar to 2020, AN distinctive activity is undoubtedly influenced by both the weather and agricultural techniques. Throughout the time between fertilisation and sowing, the weather was humid resulting in the potential leaching losses from liquid based AN. Additionally, compared to the other BBFs, the AN application appeared to result in higher salt-induced stress, as observed in plant response in the field. However, this was not clearly reflected in the soil EC measurements, which did not show consistently elevated values in the AN-treated plots. The EC of AN was the greatest of all the products, but the amount of AN applied was the lowest of all products since it contains highest % of N. Considering the varying effects of AN observed in two different years of field trials, there is a possibility of rapid volatilisation after application. AN may volatilise more due to its weaker soil binding compared to AS (Fenn and Hossner, 1985). The sulphate in AS enhances ammonium retention and acidifies the microsite, reducing NH₃ loss (Fenn and Hossner, 1985). In contrast, AN dissolves rapidly and may locally increase pH, favouring volatilisation under certain conditions (Black et al., 1985). This could offer an explanation for the diverse outcomes observed in the field, where the volatility of AN may have led to different impacts on the crops or soil conditions in each trial year.

Continuing the pattern observed in yield in 2022 silage maize field trials, high standard deviations were evident, particularly in the case of AS at 40 % and synthetic fertilisers at 100 %. This variance may be attributed to an emergency harvest, where the supplied N to the silage maize was not fully utilised for plant growth (Zhao et al., 2015). A similar result was observed by other studies, where the limitation of water resulted in lower N uptake in stem and grain for silage maize (Hussain et al., 2019; Hammad et al., 2017). Furthermore, the accelerated N uptake, especially in silage maize, occurs during later reproductive stages (after 80-110 days), underscoring the impact of early harvest (Motasim et al., 2022). The NFRV and FRUE showed promising potential for AS and AN compared to synthetic fertilisers (Table 5). However, no significant differences were noted in all cases. Similar results were observed by Szymańska et al. (2019), where AS tested on silage maize showed an NFRV of 95 - 100 %, making it comparable to synthetic fertilisers. Despite AS and AN displaying NFRV values of > 100 % at a 70 % N dosage, the high standard deviations pose challenges in interpreting the results.

4.2. Environmental performance

4.2.1. Nitrate leaching risk

Despite the generally high standard deviation in all tested treatments, attributed to weather-induced variability and the variable nature of soil NO_3^- -N in N-rich fields, BBFs showed comparable performance to the synthetic N application. In 2019, the nitrate leaching risk was high, primarily because no catch crop was planted between 2019 and 2020. This decision was driven by the need for a fine seedbed in 2020 to accommodate spinach, which requires finely prepared soil for optimal growth. Additionally, the very sandy soil increased the risk of nitrate leaching, as its low water retention exacerbated nutrient loss.

For 2020 spinach, significant differences were observed when comparing both products and dosages (for AS at 100 %). During harvest, the majority of nitrate in the soil was concentrated in the topsoil layer, suggesting minimal leaching risk had occurred. For instances for NPK, AS, and AN, a relatively high amount of ammonium was measured in the topsoil layer at harvest (details in Annex). This could imply a reduced nitrification for the treatments, which could be a possible reason for reduced N uptake and higher N residue. Additionally, the residual nitrate content was lowest in the negative control and PK. The maximum residual nitrate was observed for AN at 100 % of the N dosage,

significantly higher than all other treatments. This might be attributed to fluctuations in weather conditions (sandstorms), which could result in low N uptake and higher nitrate residue for plots treated with AN. However as compared to 2019, nitrate leaching risk reduced due to the establishment of maize as a catch crop, absorbing a substantial amount of N.

For 2021 early potatoes, the residual nitrate in the soil profile (0–90 cm) during the post-harvest period did not significantly affect the leaching risk for BBFs in comparison to NPK. However, the nitrate residues from all of the treatments (including the negative control) were comparatively high (Fig. 3). According to Flemish environmental guidelines of 2021, nitrate residue shall not exceed the upper limit of 90 kg NO₃-N ha⁻¹ (depending on the location of the field) (Vlaamse, 2021). Because early potatoes can only absorb 50–60 % of the supplied N and have shallower roots (Muleta and Aga, 2019), significant nitrate residue is often seen as being prevalent in the case of potatoes (Hendriks et al., 2021; Jiang et al., 2022). Additionally, the effect of rainfall throughout the growing season is inversely related to the nitrate residue in the top 0–90 cm of soil (Fig. 1).

Similar to the findings in 2021, in 2022 silage maize, nitrate residues did not significantly increase the leaching risk for BBFs compared to NPK, although overall residues remained high (Fig. 3). Specifically, in the case of AN at 100 % N dosage, the residual nitrate was found to be the maximum with a high standard deviation in all treatments. The primary reason for this elevated nitrate level in the winter period is attributed to the emergency harvest conducted nearly 1.5 months before the completion of the crop cycle. This premature harvest led to improper uptake of N during the growth period. These results can be corroborated with N uptake data (Fig. 2), where all treatments and N dosages exhibited nearly equal N uptake. Additionally, maize is known to have higher N uptake in the final stages of its crop cycle (Motasim et al., 2022), which also explains the higher N residues observed in this case.

4.2.2. Gaseous emissions

4.2.2.1. N₂O emission. The synthetic fertilisers UREA (0.15 %) and CAN (0.12 %) produced maximum levels of N₂O emissions among the treatments under study, followed by those from BBFs: AS (0.10 %) and AN (0.08 %). In contrast to synthetic fertilisers, BBFs derived from primary and secondary processing of manure/digestate are anticipated to result in lower N2O emissions (Fig. 4a). BBFs release N more gradually than synthetic fertilisers, reducing the accumulation of nitrate in soil that fuels N2O emissions (Castejón-del Pino et al., 2024). Their organic content supports microbial processes that favour complete denitrification ($N_2O \rightarrow N_2$), further lowering emissions (Wei et. al., 2024). Additionally, BBFs buffer soil conditions, avoiding the sharp pH or osmotic changes linked to higher N2O production (Lazcano, Zhu-Barker and Decock, 2021). A similar result is observed by Shrivastava et al. (2023) and Egene et al. (2022), where ammonium hydroxide based BBF showed lower emissions compared to synthetic N fertilisers. This phenomenon is associated with the rapid hydrolysis occurring in the soil within hours of application, leading to increased NH₄ availability, subsequent nitrification, and N2O production (Van der Weerden et al., 2016). The increased N2O emissions from CAN compared to BBFs could also be influenced by soil texture as it can lead to sustained high WFPS (Harty et al., 2016). Furthermore, the elevated N2O emissions observed in urea and CAN may be attributed to a combination of direct N2O production resulting from NH₄ nitrification and indirect N₂O release induced by NH_3 oxidation (Egene et al., 2022).

 N_2O emissions from pig slurry, could also be due to the availability of organic carbon (OC), serving as an energy source for denitrifying bacteria (Egene et al., 2022). This heightened activity reduces soil oxygen levels, promoting the denitrification of initially nitrified NH_4^+ in the fertilisers and releasing more N_2O (Hendriks et al., 2021; Velthof and Rietra, 2019). Another theory describing the reduced emissions for AS

and AN could be effect of reduced pH of products on nitrate reductase (Shrivastava et al., 2023; Wang and Li, 2019). It should be noted that the peak in N_2O emissions is likely attributed to nitrification, recommending a more extended duration experiment for a comprehensive understanding of the emissions pattern.

4.2.2.2. CO2 and CH4 emissions. For CO2 emissions, the initial OC content of the BBFs and synthetic fertilisers directly affects the CO₂ emissions from the mesocosms (Fig. 4b). The absence of OC in AS and AN resulted in insignificant emissions from these BBFs. Consequently, the cumulative emissions exhibited a negative trend for products with minimal carbon content (Fig. 4b). Similar findings have been reported in other studies (Egene et al., 2022; Hendriks et al., 2021; Shrivastava et al., 2023), where BBFs with negligible OC displayed either no emissions or negative cumulative emissions. The primary source of CO₂ flux in the soil stems from the respiration of soil microbes and decomposed plant roots (Xu and Shang, 2016). Consistent with this trend, UREA and pig slurry, characterised by substantial OC content, demonstrated emissions constituting 8.97 % and 5 % of the total carbon contribution from the respective products. Organic fertilisers, due to their elevated soil OC content that promotes microbial respiration, typically exhibit significantly higher emissions (Bednik et al., 2023; Egene et al., 2022). The carbon released from BBFs in soils is categorised as biogenic carbon, resulting in a C-neutral status. Any observable CO2 emissions in these products are attributed to the positive priming effect of the soil pre-existing carbon (Kuzyakov et al., 2000).

Throughout the entire duration of the experiment, cumulative methane (CH₄) emissions from all investigated treatments were lower than those from the negative control. This led to negative cumulative emissions when the emissions from the bio-based fertilisers (BBFs) were subtracted from those of the negative control treatment (Fig. 4c). This negative trend in cumulative emissions can be attributed to the presence of aerobic conditions during the incubations, resulting in consistently low CH₄ emissions throughout the experiment. Additionally, the application of manure-derived products to the soil was found to enhance soil aeration, subsequently reducing CH₄ emissions (Egene et al., 2022; He et al., 2023). Furthermore, measurements revealed a high degree of variability in flux, and consequently, no significant difference (p < 0.05) was observed among the treatments. The net soil CH₄ flux is an outcome of methanogenesis and methanotrophism (Yu et al., 2023). All BBFs and synthetic fertilisers (CAN and urea) induce negative methane emissions from the soil, as their CH₄ uptake exceeds their CH₄ production.

The present study was conducted under variable and, at times, challenging weather conditions, which may have influenced crop yield responses and nitrate leaching dynamics across years. The scope of the trial was restricted to a limited number of crop types, which constrains the generalisation of results across broader crop rotations. While the short-term incubation provided controlled insights into gaseous emissions, field-scale and longer-term monitoring is required to better understand the temporal dynamics and cumulative impacts of BBFs on nitrogen emissions. In addition, future research should address the performance of ammonium salts derived from animal manure, as their composition and behaviour may differ from waste-derived AS and AN evaluated here. Such work would provide a broader evidence base for assessing agronomic effectiveness, environmental performance, and regulatory suitability of animal manure-derived BBFs in circular agricultural systems.

5. Conclusions

The present study conducted a comprehensive 4-year field trial to assess the agronomic and environmental impact of AS and AN as BBFs and their potential to serve as alternatives to synthetic N fertilisers. Across all years, BBFs, particularly AS, demonstrated performance comparable to synthetic N fertilisers in terms of yield, N uptake, and

NFRV. In the unique conditions of 2021, with sandy soil and wet, cold weather, the application of BBFs and synthetic fertilisers before ploughing (with BBFs injected at a depth of approximately 20 cm before planting) contributed to substantial mineral N losses. This leaching risk, more pronounced in areas with higher N application dosage and where a greater percentage of N was available in mineral form earlier in the season. However, this factor did not exhibit a significant difference in leaching risk between BBFs and synthetic fertilisers across all years.

In general, the field trial results highlighted the significant influence of climatic and soil conditions on overall performance. Instances of relatively low precipitation and drought conditions in 2019, 2020, and 2022 resulted in decreased yields for all treatments. Additionally, the sandy soil low water retention capacity exacerbated the impact of drought conditions, leading to consistently lower yields. This soil texture not only limited moisture availability for crops but also heightened the risk of nitrate leaching, particularly during periods of inadequate rainfall. Finally, short-term gaseous emissions analysis revealed that AS and AN exhibited the lowest emissions compared to all other treatments. This was primarily due to synthetic fertilisers quick hydrolysis, leading to nitrification, a contrast to the slower processes observed with the BBFs. To conclude, this study highlights the positive potential of BBFs, especially AS, to replace synthetic fertilisers, showcasing promising agronomic and environmental performance. Despite variations in climatic conditions, BBFs demonstrated resilience and effectiveness, reinforcing their viability as sustainable alternatives in agricultural practices.

CRediT authorship contribution statement

Nimisha Edayilam: Writing – review & editing, Supervision, Project administration, Conceptualization. Ivona Sigurnjak: Writing – review & editing, Validation, Supervision, Project administration, Conceptualization. Amrita Saju: Writing – review & editing, Visualization, Methodology, Investigation, Formal analysis, Data curation. Vaibhav Shrivastava: Writing – review & editing, Writing – original draft, Visualization, Software, Methodology, Investigation, Formal analysis, Data curation. Erik Meers: Writing – review & editing, Supervision, Resources, Project administration, Funding acquisition. Tomas Van De Sande: Writing – review & editing, Methodology, Data curation.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at doi:10.1016/j.agee.2025.110072.

Data availability

Data will be made available on request.

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