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Digestate in replacement of synthetic fertilisers: A comparative 3–year field study of the crop performance and soil residual nitrates in West-Flanders

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ABSTRACT

Nitrogen (N) is an essential macronutrient for plant growth. As a widespread source of plant-available N, ammonia synthesis via the Haber-Bosch process has proven an extremely valuable commodity in farming systems since the middle of the twentieth century. However, its heavy reliance on ever-shrinking fossil fuel reserves and its sizeable carbon footprint have fostered the exploration of alternative, more sustainable, fertilising prospects. Through the recycling and reuse of nutrient byproducts, biobased fertilisers (BBF) can help reduce the European Union's dependency on imported synthetic fertilisers. In this study, we examined digestate, the liquid fraction of digestate, pig slurry and pig urine as potential substitutes for synthetic fertilisers. In a full-scale field approach using a different crop each year (maize, spinach, potatoes), the agronomic performance of the treatments (defined as the crop N uptake and the crop yield) and the environmental performance (taken as the residual soil nitrates after harvest) of the BBF treatments were compared with those of a synthetic fertiliser benchmark (calcium ammonium nitrate) at three N regimes. As regards short-term fertilising capability, results showed that yields obtained from BBFs were not statistically different ($p > 0.05$) than those obtained with synthetic fertilisers. Likewise, for soil residual nitrates (0–90 cm), measured in October–November of each year, no difference ($p > 0.05$) was detected between the BBFs and the synthetic fertiliser reference treatments. However, the non-superiority test showed that some BBFs tended to perform better in terms of residual nitrates than the synthetic regimes. Generally, results pointed to a fast N release ability of the BBFs, indicated by the presence of nitrates at different soil depths. Hence, as with the mineral fertiliser, BBFs were prone to leaching which calls for adequate N management strategies. The N content of some BBFs were shown to vary over time, hence adequate and timely nutrient characterisations must be carried out prior to field application to ensure a more accurate N accountancy and reduce risks of over-fertilisation (or under-fertilisation).

1. Introduction

In the aftermath of World War II, agriculture's main emphasis was on improving crop production. During the ensuing Green Revolution, food supplies were greatly increased with the advent of short-stemmed high-yielding strains that responded positively to synthetic fertilisers and irrigation (Mazoyer and Roudart, 2002; Phillips, 2014). This tremendous advance in agricultural productivity to support population growth was due in no small part to ammonia (NH_3) fertilisers that were being manufactured via the Haber-Bosch process at an industrial scale (Erisman et al., 2008). So much so that, in a little less than a century, reactive nitrogen (N) production from the Haber-Bosch process quickly established itself as a central cog in the global agrifood system, seeing that its production grew by leaps and bounds to reach 100 Tg N y^{-1} by 2000, of which 85 % was appointed to fertilisers (Galloway et al., 2003). In the European Union, wheat yield per unit of land more than doubled from 1961 to 2021 (Food and Agriculture Organization of the United Nations (FAO), 2023a). In this sixty-year timespan, European imports of synthetic N fertilisers increased by an estimated factor of 13.5 while at the same time the agricultural use of these fertilisers almost tripled (Food and Agriculture Organization of the United Nations (FAO), 2023b). However, the rapid escalation of synthetic N inputs for intensive

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crop and livestock production has led to substantial losses of reactive N (NO_x, NH_3, N_2O, NO_3^-) to air, water, and soil with detrimental consequences to the environment (Garnier et al., 2023; Velthof et al., 2014). Concomitantly, diminishing returns from N fertilisation over time indicate that the N efficiency of cereal production has dropped from roughly 80 % in 1960–20–30 % in 2000, while about 50–70 % of the applied N was lost to the environment (Tilman et al., 2002). To further compound the issue, population growth (mainly in developing countries) is projected to bring about an increase in total global food demand of 35–56 % between 2010 and 2050 (van Dijk et al., 2021) which will more than likely put an additional strain on the current agrosystem's heavy reliance on synthetic fertilisers (Rizzioli et al., 2023). Problematically, as dominant as the Haber-Bosch process has become for the production of NH_3 fertilisers, it accounts for 1–2 % of global energy consumption and 1.44 % of CO_2 emissions worldwide (Kyriakou et al., 2020).

In an attempt to improve agricultural efficiency and, at the same time, cut back on the negative impacts on air, soil and water resources, several complementary strategies are being put forward such as sustainable intensification (Cassman and Grassini, 2020; Pretty and Bharucha, 2014), high-precision agriculture and nutrient recovery (Anlauf, 2023; Wali et al., 2022). In particular, nutrient recovery from waste streams proposes an alternative to the energy-and-carbon-intensive Haber-Bosch-manufactured fertilisers, the prices of which are directly linked to those of natural gas (Mehta et al., 2015). Recent supply chain disruptions and limited access to natural gas have led to a surge in synthetic fertiliser prices, dramatically increasing production costs and food prices (Alexander et al., 2023). These events, combined with the depletion of fossil-fuel reserves (Welsby et al., 2021), have spurred a renewed interest in the circular economy and organic waste valorisation to produce biobased fertilisers (BBFs) (Chojnacka et al., 2022; Egan et al., 2022). In this context, anaerobic digestion is presented as having the potential to alleviate some of these environmental challenges in reason of the positive externalities it allows within a circular economy framework (Burg et al., 2023; Srinivasan, 2008). One of the main appeals of this technology is the capacity to recover energy from various biomass inputs — slurries, residues from the agrifood industry, municipal waste, wastewater treatment sludges — to produce renewable heat, electricity, and gas (Hagman et al., 2018; Weiland, 2010) thereby reducing the corresponding amount of fossil fuels for these applications (O'Shea et al., 2020) and resulting in avoided emissions from the decomposing feedstocks that are confined in a sealed digester (Afotey and Sarpong, 2023). Another asset, which comes more directly into consideration for the present study, is the biogas slurry — also called digestate — that is generated as a co-product once the anaerobic digestion process has run its course. Indeed, digestate retains all the initial nutrients contained in the waste streams, making it an inexpensive and readily available BBF (Oldani et al., 2023; Pastorelli et al., 2021). It also paves the way for targeted resource recovery strategies of macronutrients and a streamlined management of nutrient-rich byproducts (De Vrieze et al., 2019; Meers et al., 2020). Moreover, during the anaerobic digestion process, the organic N contained in the initial feedstocks undergoes a partial mineralisation thereby increasing the total share of ammonium N (NH_4^+–N), a form that is readily available for uptake by plants (Cavalli et al., 2016; Möller and Müller, 2012). For these reasons, digestate is being investigated as a more sustainable alternative to Haber-Bosch fertilisers for crop production (Aso et al., 2022; Baştabak and Koçar, 2020; Reuland et al., 2022). Importantly, 85 % of the rock phosphate reserves — an indispensable non-renewable resource for the production of phosphorus (P) fertilisers — is concentrated in the hands of Morocco, China, Syria, Algeria (Brownlie et al., 2023). In response, the European Union has acknowledged that phosphate rock, of which it only has very small deposits, is subject to high supply risk and has accordingly placed the strategic resource on its list of Critical Raw Materials (Smol, 2019). In view of the depletion of this element, circular P recovery strategies are necessary to increase resilience and ensure food security (Vu et al., 2023).

The main objective of the present study was to assess the potential of BBFs as viable substitutes to synthetic fertilisers and, in a larger scheme, explore to what extent a full-scale fertilisation campaign is able to break away from the linear *modus operandi* in favour of a more circular agriculture using BBFs as the sole source of N-input. To this aim, the effects of four BBFs (digestate, the liquid fraction of digestate, pig urine and pig manure) were compared with a reference synthetic fertiliser (calcium ammonium nitrate) at three application rates over the course of a 3–year field study. The specific metrics that were evaluated were (i) the agronomic performance of the treatments, defined as the crop N uptake and the crop yield, (ii) the environmental performance, taken as the residual soil nitrate N (NO_3^-–N) after harvest. As regards these performance metrics, it was hypothesised that the use of BBFs would not induce significant differences in comparison with the synthetic reference.

2. Materials and methods

2.1. Experimental design

2.1.1. Site description and field screening

The 3–year study was conducted on sandy soil in a field located in Wingene (51°02'09.4"N 3°10'42.4"E) in the Belgian province of West Flanders. The field was part of a mixed farming system with a combination of vegetable crops and cattle. Before the trials, it was customary to apply manure annually at a rate of 50 t ha^{-1}. Before tillage, a preliminary screening of the field was carried out to minimise spatial variability. To this effect, the useable surface of the field was divided into 39 sections (15 ×18 m) and areas exposed to frequent traffic (sprayer tracks) as well as field margins were excluded. The sections were then screened for disparities via three strategies: (i) multispectral Normalized Difference Vegetation Index (NDVI) images were taken (Micasense camera) to detect variations in the biomass of the preceding Italian ryegrass catch crop; (ii) penetrologger measurements to detect soil compaction and resistance to penetration (penetration speed 2 cm sec^{-1}); (iii) soil sampling in late March and mid–April 2019 to determine general soil fertility levels. Following these assessments, 3 of the 39 sections, where the regrowth of the Italian ryegrass was visibly impaired (Fig. S1), were discarded. The penetrologger probing campaign (10 measurements per section) revealed the presence of a plough pan at a depth of 30–40 cm, where penetration resistance increased sharply across the entire field (Fig. S2). Four blocks were thus delineated, each regrouping plots with similar soil penetration resistance characteristics. On the east side of the field (blocks I and II), penetration resistance was the highest, averaging 8 Mpa. With an average 6 MPa, there was less compaction on the west side (blocks III and IV) and penetration resistance below 40 cm even dropped in the southwestern area (block IV). The resulting plots were spread across the 36 selected sections and four blocks in a randomised complete block design (RCB). The georeferenced gross plots measured 48 m^2 (6 ×8 m) each, inside of which a net plot (6 ×4 m) was delineated for sampling and harvesting. Each plot received the same treatment systematically over the span of the 3–year experiment (Fig. S3). The average physicochemical properties of the topsoil across the four blocks, sampled shortly before sowing were: pH KCl = 5.7; EC = 55 mS cm^{-1}; NH_4^+–N = 12 kg ha^{-1}; NO_3^-–N = 17 kg ha^{-1}; total organic carbon (TOC) = 0.8 % on dry weight (DW); total nitrogen (TN) = 71 mg 100 g^{-1} DW; P = 66 mg 100 g^{-1} DW; potassium (K) = 24 mg 100 g^{-1} DW; magnesium (Mg) = 12 mg 100 g^{-1} DW; calcium (Ca) = 81 mg 100 g^{-1} DW; sulphur (S) = 1.5 mg 100 g^{-1} DW, sodium (Na) = 0.5 mg 100 g^{-1} DW.

2.1.2. Crop rotation and monitoring

The previous crops were maize (2016 and 2017) and potatoes (2018). In 2016 and 2018, a catch crop of Italian ryegrass was installed after the main crop, after the main crop the soil was left bare in 2017. For this study, the crop rotation was as follows: maize (*Zea mays* cv. Telias) in 2019 (year 1); spinach (*Spinacia oleracea* cv. Spirico) in 2020 (year 2); first early seed potatoes (*Solanum tuberosum* cv. Anosta) in 2021 (year 3). For maize, the sowing density was 95,000 ha^{-1} with a row spacing of 75 cm. For spinach, the sowing density was 3 million ha^{-1} with a row spacing of 12.5 cm. The potatoes were planted every 37.5 cm with the ridges spaced 75 cm apart. In year 1, the soil was left bare after the maize harvest. In year 2, the spinach harvest was followed by silage maize. In year 3, the potatoes were followed by Italian ryegrass. The field was subjected to conventional ploughing and levelling with a rotary harrow before sowing, except for year 2 (spinach) where non-reversing soil tillage was adopted. At separate moments in time, remote-sensed NDVI images were acquired as a complementary means to assess crop vigour (Table 1).

2.1.3. Product sourcing and fertilisation regime

The studied BBFs were pig urine (PU), pig slurry (PS), digestate (D) and the liquid fraction of digestate (LFD). For comparison purposes, a conventional mineral fertilisation treatment using calcium ammonium nitrate (CAN) was used as a benchmark. The BBFs were transported in tanks from local farms and manure-processing plants — all situated in the province of West Flanders in the Flemish Region of Belgium — and delivered onsite where they were transferred to caged IBC totes (1000 L). As the nutrient composition of the BBFs was subject to change over time, the products were sampled a first time on-farm for chemical characterisation a few weeks ahead of the trial, prior to transportation. The LFD came from the AM Power biogas plant in Pittem. It has a treatment capacity of 180 kt y^{-1} (89 % food waste, 8 % manure and 3 % maize) spread over five digesters (20,000 m^3) which produce 7.5 MW of electricity. It is a thermophilic digestion with a retention time of 50–60 days and 10 days in the post-digester. The LFD was obtained from digestate (9 % DM) which was centrifuged after the addition of a polymer for coagulation and flocculation. The PS was from a small farm located in Zwevezele which houses approximately one hundred pigs. The pig stable was equipped with a slatted floor and a slurry pit underneath, from which the PS was taken. The PU came from a pig farm situated in Staden. A manure separation system (VeDoWelfare system) located beneath the slatted floor, and equipped with a manure scraper which runs across the slanted collection gutter, allowed urine (PU) to percolate into a separate collection channel at the very bottom. The D product was collected from a thermophilic (45 °C) biogas plant in Hooglede. The AD plant has a treatment capacity of 45,000 t y^{-1} and a retention time of 40 days. The feedstocks are 60 % animal manure and 40 % biowaste (vegetable waste). On the day of the field fertilisation, each BBF was transferred back and forth from the IBC storage tote to the tractor's tank to achieve thorough homogenisation (Fig. S4). At that point, a sample was taken to determine the exact concentrations of nutrients at the time of application (Table 2). This sampling of BBFs at different points in time explains the variations between the targeted N value and the applied amounts of BBFs in certain cases.

The optimal recommended N fertiliser requirements, corresponding to a dosage of 100 %, were determined at the beginning of each season by the Provincial Advice Centre for Agriculture and Horticulture (Inagro, Rumbeke-Beitem, Belgium) according to the crop requirements and the levels of residual N in the soil before sowing. Based on this optimal dosage, the fertilisers (CAN, PU, PS, D, and LFD) were applied at three different regimes: a low dosage corresponding to 40 % or "low" hereafter; a medium dosage of 70 % or "med" hereafter and 100 % of the recommended N dosage or "high" hereafter. The different amounts of N, P and K applied are listed in Table 3. Each regime (low, med, and high) was applied in quadruplicate (one per block); thus, twelve plots were allocated per fertiliser treatment, resulting in a total of sixty fertilised plots (3 dosages x 4 replicates x 5 fertiliser treatments). Additionally, eight replicates of an unfertilised control (labelled 'UNF_CL'), as well as a PK treatment receiving mineral PK but no added N (labelled 'PK_CL') were included, bringing the final number of plots to 76 for the experiment. To the exception of UNF_CL, all treatments supplied additional P, K and S in slight excess of crop needs to prevent nutrient deficiencies so as to better ascertain the effect of N on plant yield. This complementary fertilisation was adjusted by taking into account the quantity of these elements already present in the BBFs. In this manner, CAN (30 % N) (for the synthetic reference treatments), triple superphosphate (46 % P_2O), patentkali (30 % K_2O|42 % SO_3|10 % MgO), haspargit (23 % K_2O|22 % SO_3) and potassium chloride (60 % K_2O) were used in combination, each according to needs, to reach the final targets.

For the maize trial (year 1), BBFs were applied on April 24th and the mineral N fertiliser on April 30th, followed by ploughing on May 1st and sowing the day after. For the spinach campaign (year 2), all fertilisers were applied on the same day (March 24th). The sowing took place on April 13th. For the potatoes (year 3), the fertilisers were applied over three consecutive days (23–25 March) immediately followed by ploughing. Potatoes were planted on March 27th (Table 1). The application of the different products was conducted using an experimental

Table 1
Activity log of field trial.

2019		
	15–Feb	Screening: drone imagery remote sensing
	19–Feb	Screening: penetrologger
	28–Mar	Screening: topsoil sampling for chemical analysis
	16–Apr	Screening: topsoil sampling per block for chemical analysis
	24–Apr	Application of BFFs
	30–Apr	Application of mineral N (on gross plots)
	01–May	Ploughing
	02–May	Field preparation (rotary harrow) and sowing
	10–May	Application of mineral P and K (on gross plots)
	10–May	Application of mineral CAN outside of gross plots
	10–May	Visual evaluation emerging sprouts
	26–Jun	Evaluation of crop growth: drone imagery remote sensing
	07–Aug	Evaluation of crop growth: drone imagery remote sensing
	18–Sep	Harvest outside of gross plots
	19–Sep	Harvest, yield determination and sampling of crops (block IV)
	25–Sep	Harvest, yield determination and sampling of crops (blocks I–III)
	14–Oct	Determination of residual nitrate: soil sampling (0–90 cm)
	29–Oct	Incorporation of stubs and levelling of field
2020		
	25–Feb	Soil sampling per plot for determination of mineral N
	24–Mar	Application of BBFs
	24–Mar	Application of mineral N and P
	26–Mar	Application of mineral K and S (haspargit)
	13–Apr	Sowing
	29–Apr	Drone imagery remote sensing
	07–May	Visual evaluation of crops
	10–May	Strong winds and damage to the young plants (dust)
	10–May	Irrigation to counter wind damage (10 L/m^2)
	13–May	Drone imagery remote sensing
	15–May	Irrigation (25 L/m^2)
	26–May	Irrigation (25 L/m^2)
	01–Jun	Visual evaluation of crops
	02–Jun	Drone imagery remote sensing
	02–Jun	Harvest, yield determination and sampling of crops
	03–Jun	Determination of residual nitrate: soil sampling (0–90 cm)
	26–Sep	Harvest of maize (catch crop)
	28–Oct	Determination of residual nitrate: soil sampling (0–90 cm)
2021		
	01–Mar	Soil sampling per plot for determination of mineral N (0–90 cm)
	23–Mar	Topsoil sampling per block for chemical analysis
	23–Mar	Application of BBFs
	25–Mar	Application of mineral N, P and K
	26–Mar	Ploughing
	27–Mar	Field preparation (rotary harrow) and sowing
	25–May	Visual evaluation and emergence assessment (number of stems)
	28–Jun	Visual evaluation
	02–Aug	Determination of remaining aboveground biomass per plot
	04–Aug	Yield determination (tubers)
	08–Aug	Determination of residual nitrate: soil sampling (0–90 cm)
	18–Aug	Ploughing and sowing of Italian ryegrass
	10–Nov	Determination of residual nitrate: soil sampling (0–90 cm)

Table 2
Main physicochemical properties of the biobased fertilisers used during the field trials.

		2019				2020				2021			
		PS	PU	D	LFD	PS	PU	D	LFD	PS	PU	D	LFD
pH		/	/	/	/	8.1	8.2	8.2	8.1	8.2	7.9	8.3	7.9
EC		/	/	/	/	41	43	42	42	42	25	29	30
DM	g kg^{-1} FM	118	28	105	56	145	34	90	60	116	17	79	34
NH_4^+–N	g kg^{-1} DM	41	150	25	52	41	151	34	65	40	149	29	90
Kjeldahl–N	g kg^{-1} DM	68	177	53	79	62	183	67	99	65	178	65	133
C_{org}	g kg^{-1} DM	379	311	316	293	428	317	298	280	394	296	333	310
OM	g kg^{-1} DM	682	559	569	527	771	571	536	505	709	534	600	557
P	g kg^{-1} DM	19	12	24	23	18	12	25	24	20	7	23	15
K	g kg^{-1} FM	40	109	23	41	40	116	30	44	40	116	24	73
NO_3, NO_2	g kg^{-1} DM	0	0	0.3	0.2	0	0	0	0	0	0	0	0
C/N	/	5.6	1.8	5.9	3.7	6.9	1.7	4.5	2.8	6.1	1.7	5.1	2.3

Table 3
Fertilisation regime of applied N, P_2O_5 and K_2O (kg ha^{-1}). Under the N column, the amount that was applied (left side) and the target value (right side, in between parentheses) are presented side–by–side.

	Year 1			Year 2			Year 3		
Treatment	N	P_2O_5	K_2O	N	P_2O_5	K_2O	N	P_2O_5	K_2O
UNF_CL	/	/	/	/	/	/	/	/	/
PK_CL	/	108	250	/	133	330	/	118	323
CAN_low	60 (60)	108	250	84 (84)	133	330	56 (56)	118	323
CAN_med	106 (106)	108	250	147 (147)	133	330	98 (98)	118	323
CAN_high	151 (151)	108	250	210 (210)	133	330	140 (140)	118	323
PS_low	42 (60)	108	250	87 (84)	53	393	48 (56)	114	358
PS_med	71 (106)	108	250	152 (147)	93	440	84 (98)	112	385
PS_high	102 (151)	108	250	217 (210)	133	487	120 (140)	109	411
PU_low	69 (60)	108	250	83 (84)	141	392	52 (56)	111	364
PU_med	123 (106)	108	250	145 (147)	146	438	92 (98)	105	395
PU_high	178 (151)	108	250	207 (210)	152	484	131 (140)	91	426
D_low	82 (60)	108	250	80 (84)	171	370	55 (56)	116	348
D_med	137 (106)	108	250	140 (147)	199	400	97 (98)	115	366
D_high	197 (151)	108	250	200 (210)	227	431	138 (140)	114	385
LFD_low	75 (60)	108	250	83 (54)	160	373	55 (56)	116	360
LFD_med	126 (106)	108	250	145 (147)	181	404	97 (98)	114	387
LFD_high	180 (151)	108	250	208 (210)	201	436	138 (140)	113	414

UNF_CL: unfertilised; PK_CL: synthetic PK; CAN: calcium ammonium nitrate; PS: pig slurry; PU: pig urine; D: digestate; LFD: liquid fraction of digestate. Under the 'Treatment' column, suffixes low; med and high correspond to 40; 70 and 100 % of the recommended optimal N dosage, respectively.

vehicle that was custom designed to administer both viscous slurries and highly fluid mineral fertilisers (Fig. S4). This hybrid machine was equipped with a vacuum pump and a coulter injection system with light duty harrow discs adapted for viscous fertilisers (accuracy of 0.2 t ha^{-1}), and a hose pump connected to a network of tubes for mineral fertilisers (accuracy of 10 kg ha^{-1}). Immediately after a plot was fertilised, the product was incorporated with rotary blades and the soil was sealed with a roller to ensure that ammonia volatilisation from NH_4^+–N–rich BBFs would be kept to a minimum (Fig. S4).

2.2. *Plant harvesting and soil residual nitrate sampling*

In year 1, maize was harvested manually from a net plot surface of 24 m^2 (4 ×6 m). In year 2, aboveground biomass (spinach leaves) was harvested with a Haldrup plot combine harvester from a 12 m^2 area inside each plot (1.5 ×8 m). In year 3, the potatoes were harvested by hand at a rate of 2 ridges per plot and 6 m along each ridge (1.5 ×6 m). Belowground (tubers) and aboveground biomass (leaves) were collected separately. Fresh yields were determined onsite. At harvest, a composite soil sample was elaborated from 6 subsamples per plot taken at three depths each (0–30 cm; 30–60 cm; 60–90 cm) with an automatic soil sampler. Exceptionally, for maize, soil samples could not be taken around the time of harvest because of the weather conditions. Each year, the soil was also sampled in the same way prior to fertilisation to determine the N concentration (March 2019, February 2020, March 2021) and after the harvest, that is, at the end of the Flemish legal reference period for determining soil nitrates which spans from October 1st to November 15th. All samples (plant and soil) were transported to the laboratory where they were kept in polyethylene containers at 4 °C for further chemical characterisation.

2.3. *Recording of meteorological data*

A weather station was set up on the nearby farm less than 500 m away from the field. It monitored the temperature (hourly measurements) and rainfall (tipping bucket rain gauge). For comparison purposes, the monthly precipitation data of the Royal Meteorological Institute of Belgium (KMI-IRM) for the locality of Wingene was also collected. The annual climographs for the three growing seasons were plotted accordingly (Fig. S5).

2.4. *Physicochemical characterisations*

2.4.1. *Plant materials*

The freshly harvested biomass was weighed to determine the fresh weight (FW). The samples were subsequently placed in an oven for 24 hours at 55 °C to determine the DW, at which point they were finely milled for further chemical characterisations. The TN was determined on a PRIMACS100 Analyser series (Skalar Analytical BV, Breda, Netherlands). To determine macro-and-microelements, 5 mL HNO_3 65 % was added to 0.1 g sample and placed inside a sonicator for 30' at 50 °C after which the suspension was microwave-digested (UltraWAVE, Milestone Srl, Sorisole, Italy) and subsequently filtered (Whatman No. 43, Maidstone, UK). The elemental composition of the filtrates was determined via ICP-OES (Varian Inc., Palo Alto, CA, USA).

2.4.2. Soil

The soil samples were sieved through a 1–mm mesh. The DW and moisture content were determined on samples placed inside an oven at 105 °C for 24 hours. The TN and TOC were determined on a PRIMACS100 Analyser series (Skalar Analytical BV, Breda, Netherlands). Macro-and-microelements were measured on the ICP-OES (Varian Inc., Palo Alto, CA, USA) after hot plate digestion of 1 g soil in 2.5 mL demineralised water in a 3:1 (v/v) aqua regia solution of hydrochloric acid (HCl) 47 % and nitric acid (HNO_3) 65 %. The pH_{KCl} was determined using an Orion Star A211 pH electrode (Thermo Fisher Scientific, Waltham, MA, USA) in a 1/5 ratio (w/v) of fresh sample to 1 M potassium chloride (KCl). The electrical conductivity (EC) was measured with an Orion Star A212 conductivity meter in a 1/5 ratio (w/v) of sample to demineralised water. The suspension was placed on an orbital shaker for 60' and filtered (Whatman No. 43, Maidstone, UK) prior to the reading. Fresh samples were digested in 200 mL 1 M KCl to determine contents of NH_4^+–N and NO_3^-–N determined on a San^{++} Continuous Flow Analyser (Skalar Analytical BV, Breda, the Netherlands).

2.4.3. Biobased fertilisers

The pH_{KCl} was determined in the same way as for soil (Section 2.4.2). The EC was measured in undiluted fresh samples (Orion Star A212). The DW was determined in the same way as for soil. The NO_3^-–N was determined on a San^{++} Continuous Flow Analyser (Skalar Analytical BV, Breda, the Netherlands). For Kjeldahl N, the sample was first digested with sulphuric acid (H_2SO_4) (350–380 °C). Then, NH_4^+–N was distilled with sodium hydroxide (NaOH), the NH_3 distillate captured in a boric acid solution (H_3BO_3) and the captured ammonium ions were titrated with HCl. The P and K were determined on ashes (ICP-AES, Optima 8300, Perkin Elmer, USA) after sample incineration at 550 °C. The S was measured (ICP-AES, Optima 8300, Perkin Elmer, USA) after hot plate digestion of the aqua regia solution (HCl 47 % and HNO_3 65 %). The DW and moisture content were determined on samples placed in an oven at 105 °C for 24 hours.

2.5. Nitrogen use efficiency and replacement value

The nitrogen use efficiency (NUE) and the nitrogen fertiliser replacement value (NFRV) were used as metrics to estimate the effects of a given N fertiliser on the N uptake of the crop. To achieve this, the NUE of a considered treatment was corrected with the N uptake from the control having not received any N. The NFRV was then calculated as the ratio between the NUE of a BBF treatment and that of the conventional synthetic N treatment for a given dosage. For each fertilisation regime (40, 70, and 100 % of the recommended dosage), the conventional fertilisation was considered 100 % efficient or in other words as having an NFRV ratio of 1. It follows that NUE and NFRV were defined as (Huygens et al., 2020):

$$NUE(\%) = \frac{NU_{fertilised} - NU_{control}}{TN_{applied}} \tag{1}$$

Where $NU_{fertilised}$ is the N uptake of the crop by the N-fertilised treatment (BBF and synthetic fertilisers); $NU_{control}$ is the N uptake of the crop by the control treatment without any addition of N (corresponding to the PK_CL treatment); $TN_{applied}$ is the total amount of N applied by the considered treatment.

$$NFRV = \frac{(NU_{BBF} - NU_{control}) \ x \ TN_{BBF}^{-1}}{(NU_{CAN} - NU_{control}) \ x \ TN_{CAN}^{-1}} \tag{2}$$

Where NU_{BBF} is the N uptake of the crop from the biobased fertiliser treatment; $NU_{control}$ is the N uptake of the crop from the control treatment without N (corresponding in this case to PK_CL); TN_{BBF} is the total N applied using the considered biobased fertiliser; NU_{CAN} is the N uptake of the crop from the synthetic nitrogen treatment; TN_{CAN} is the total N applied with the synthetic treatment (calcium ammonium nitrate).

2.6. Statistical analysis

The statistical handling of the data was conducted on the SAS 9.4 software package (SAS Institute Inc., Cary, NC, USA). The datasets were first tested for normal distribution and homogeneity of variance. Subsequently, the statistical significance of the fixed effects was determined via analysis of variance (ANOVA) using the linear mixed model procedure (PROC MIXED) with restricted maximum likelihood (REML). The number of degrees of freedom was calculated using the Satterthwaite approximation method. Where significant ($p < 0.05$) treatment effects were found, pairwise comparisons were carried out with the Tukey-Kramer adjustment for multiple comparisons. The homoscedasticity of the scaled residuals was assessed graphically. The tested fixed effects were the product (type of fertiliser), the dosage of the fertiliser and the product*dosage interaction, and the block was accounted for as random effect. The separate variables under consideration were yield, N uptake and residual soil nitrates. Regarding yield in particular, results can be expressed both on FW and DW. As the outcome of the statistical analyses was very similar based on either of these, we referred systematically to the latter hereafter for the purpose of conciseness and overall clarity (statistical significance of fresh yield were still included, Table S1).

Multifactor effect size statistical computing ($p < 0.05$) was performed using R environment version 4.2.2 (R Core Team) via the RStudio integrated software version 2023.06.1 build 524 (RStudio, Inc., Boston, MA, USA). The effects of treatments (product and dosage) on dry yield and N uptake were estimated using a test of non-inferiority, and the effects on residual nitrates were estimated with a test of non-superiority. Hence, the non-inferiority tests aimed to verify whether yields and N uptake of BBFs were not unacceptably worse than a widespread reference product already in use (synthetic fertiliser). Likewise, the non-superior test aimed to verify that soil residual nitrates from BBFs would not increase in comparison with a widespread reference product already in use (synthetic fertiliser). It follows, that these tests, the unfertilised controls were disregarded to increase the statistical power of the cross-comparison between BBFs and the synthetic fertiliser. Among the CAN synthetic benchmark fertilisers (low, medium, and high), CAN_high tended to have the highest values (dry yield, N uptake and residual N). Therefore, CAN_high was taken as the reference against which the BBF treatments were compared to calculate the probability of these treatments of being above or below the synthetic benchmark. To account for field variability, a practically meaningful effect size, or tolerance value, was set at 15 % of the CAN_high mean value for dry yield, N uptake and residual N, respectively.

3. Results and discussion

3.1. Agronomic performance: biomass yield and N uptake

3.1.1. Maize (year 1)

The post-hoc cross-comparison tests did not reveal any significant contrasts between treatments (Table S3). As observed in Fig. 1, there is no discernible incremental dose-response trend. Of note, LFD_low was associated with the highest yield of all treatments with 17.3 t ha^{-1} (Table S2). While LFD_low was in fact 25 % above the intended N target (Table 3), the resulting 75 kg N applied still positioned it below LFD_med (126 kg N) and LFD_high (180 kg N) in terms of applied N for comparable yields, so that the overfertilisation would not explain why the yield and N uptake were so exceptionally high with LFD_low (data variability was particularly high). In comparison, D_high provided 2.6 times the amount of N provided by LFD_low (Table 3) for a statistically comparable yield. In the same vein, the three PS treatments fell short of the mark by 30 % in comparison with the initially intended N targets (Table 3). In spite of this apparent under-fertilisation with PS

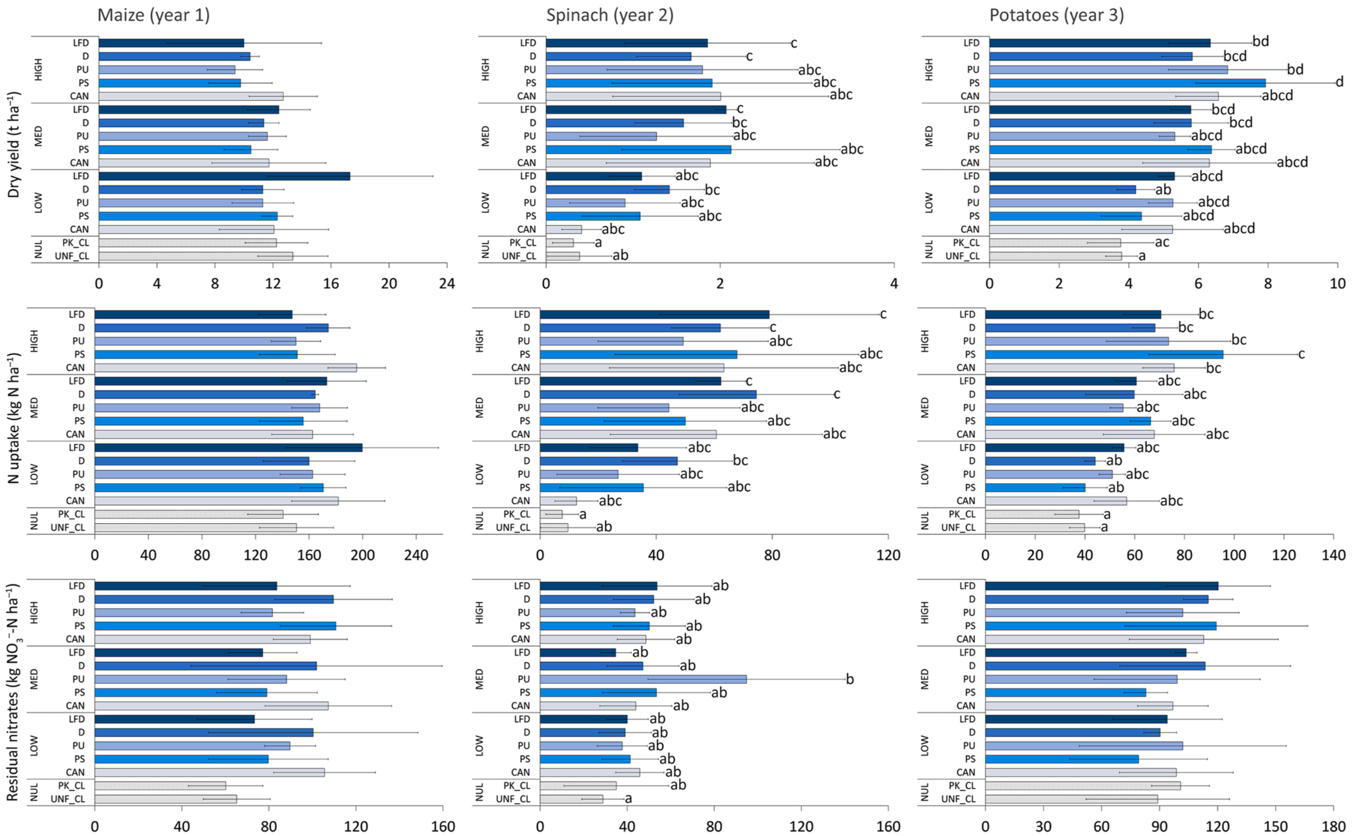

Fig. 1. Treatments and their corresponding dry yield (top), N uptake (middle), and soil residual nitrates in the first 90 cm (bottom). HIGH: 100 % of recommended N requirement; MED: 70 %; LOW: 40 %; NUL: 0 %. Means that share the same letter are not significantly different from each other ($p > 0.05$).

treatments, the yields did not seem to have been impacted significantly either way, nor did the N uptake (Fig. 1). In some cases, the average yields from the unfertilised control treatments (PK_CL and UNF_CL) even surpassed some of the N treatments, even at the highest N regimes (Fig. 1 and Table S2). This pattern could suggest that as soon as the medium and high N regimes were reached, the additional N did not benefit the plant. As sound as this statement might appear, it is important to note that the particular field conditions led to often high standard deviations for the estimated NUE and NFRV values (Eq. 1) which were often on par with, if not higher than, the calculated average (Table S2). As an example of this, the NUE dropped from 79 ± 76 % to 26 ± 23 % to 4 ± 45 % for LFD_low, LFD_med and LFD_high, respectively (Table S2). The lack of significant differences between the N uptake of the controls and the BBFs carried over to the NUE and NFRV. Hence, the interpretation of the NUE and NFRV results calls for caution. The effect size statistics for maize dry yield showed that the only standout treatment was LFD_low with a 98 % probability (1 – p, Table S6) of being non-inferior to the CAN_high reference (Fig. S6 **A**). As a yardstick, the average yield of maize silage in the Flemish region in 2019 was 40.7 t ha^{-1} (Belgian statistical office (Statbel), 2023) against 31.8 t ha^{-1} (excluding unfertilised plots) in this study. Hence a non-negligeable difference of 22 % tends to indicate that yields were generally suboptimal, even for the highest performing treatment which, paradoxically, was from one of the low N regimes (LFD_low).

Compared with the thirty–year reference period, the cumulated annual rainfall deficit for the year 2019 was high (–263 mm), whilst monthly temperatures were also higher on average, which made for an exceptionally dry and hot season (Fig. S5). Despite these suboptimal conditions, the water shortage during the first couple of months after the start of the trial in mid–April did not negatively affect maize growth and its modest water requirements could still be met during the initial vegetative stages. The first half of June was moderately wet (despite a 13.5 mm shortage on average) which proved providential for the dry soil. The months of July and August were the driest (–60 and –56 mm of rainfall compared with the reference period). June, July, and August were also unusually hot with high temperatures of 4.8, 3.8, and 3.3 °C, respectively, above those of the reference period. The climograph (Fig. S5) points to drought conditions (low rainfall and high temperatures) for the months of July and August. At the onset of July, the maize was 60 days old, hence it had entered the silk emergence phase and would have been transitioning to kernel development, hence the 2–month drought coincided with the most critical phase for moisture requirements (Liu et al., 2022; Monteleone et al., 2022) as a result of which the crop was visibly impaired. This state of affairs was worsened by the low water holding capacity of the sandy soil.

3.1.2. *Spinach (year 2)*

None of the N treatments differed significantly and the positive dose-response statistical effect ($p = 0.0018$) was due to the presence of the lower-yielding unfertilised controls (Fig. 1). The UNF_CL and PK_CL controls had the lowest average biomass yields (0.39 and 0.31 t ha^{-1}, respectively, Table S2). Upon closer inspection, some highly significant contrasts ($p < 0.01$) were noted between some of the higher-yielding N fertiliser treatments — namely, LFD_high, D_high, LFD_med, D_med, D_low — and both UNF_CL and PK_CL (Table S3). Among the N treatments, CAN_low resulted in the smallest yield with 0.41 t ha^{-1}, less than half the second lowest yield from PU_low (Table S2 and Fig. 1). When considering the spinach yields obtained at any dosage, the BBFs were not any different ($p > 0.05$) than the CAN reference treatments (Table S3).

The above pattern carried over to N uptake where the significant effect of the dosage ($p = 0.0006$, Table S1) stemmed from differences between some BBFs and the controls. The D_med treatment resulted in a very highly significant ($p < 0.001$) difference with both PK_CL and UNF_CL (Table S2). Likewise, D_high, D_low, LFD_high, and LFD_med, showcased highly significant ($p < 0.01$) differences with PK_CL and UNF_CL (Table S4 and Fig. 1). The PK_CL and UNF_CL control treatments resulted in the lowest N uptakes with 8 and 10 kg N ha^{-1}, respectively (Table S2). The highest mean N uptake was associated with LFD_high (79 kg N ha^{-1}), while the lowest was from CAN_low (13 kg N ha^{-1}), however often-high standard deviations meant that the N uptake of the N treatments were not statistically different despite, in some cases, showcasing contrasting means (Fig. 1). As these parameters are deeply interconnected, the lack of contrasts overall between most treatments (BBF and CAN) and controls on one hand, and between BBFs and the CAN reference treatments on the other hand, greatly limited the interpretability of the NUE and NFRV results (Table S2). As an example, the highest NUE was from D_low (50 ± 24 %) and the lowest was CAN_low (6 ± 9 %), while the NRFV of PS_low and D_low clocked in at 5.5 (± 5.7) and 8.5 (± 4.1), respectively. Although not significantly different (high variability in the results), a pattern could be observed in that the low treatments, in general, led to a lower yield and N uptake than the medium and high dosages, while the medium and high dosages were not that different from each other (Fig. 1). Effect size statistics for spinach dry yield and N uptake, no BBF treatment stood out significantly as being non-inferior to CAN_high values (Table S7 and Fig. S6 **B**). To put things into perspective, the spinach yield in Belgium in the year 2020 neighboured 20.8 t ha^{-1} on average (Food and Agriculture Organization of the United Nations (FAO), 2023a) against 13.7 t ha^{-1} during the present trial (excluding the unfertilised patches), or a remarkable 34 % difference (discussed further below).

In the window that stretched from sowing to harvest in early June, the conditions were unusually dry, in particular the month of May which corresponded to conditions of drought (Fig. S5). In this timeframe, the onsite tipping bucket recorded only 12 mm of rainfall. The situation was worsened by a highly unusual dust storm, with recorded wind speeds of up to 7–8 Beaufort. The spinach was irrigated twice from a small hose reel irrigation system with 50 mm of water in an attempt to curb the damage from the sandblasting and water stress (Table 1). However, the exceptionally dry spell and wind erosion, coinciding with the plant's development at a vulnerable stage, impaired the spinach's vigour and led to the observed widespread results (Fig. 1).

3.1.3. Potatoes (year 3)

A significant effect of dosage on yield was observed ($p = 0.0001$; Table S1). The pattern was similar to year 2 in that highly significant contrasts ($p < 0.01$) arose between some of the higher-yielding N treatments and the unfertilised controls (Table S3). Among the N treatments, there was a highly significant difference ($p < 0.01$) between the highest yield, obtained from PS_high, and the lowest from D_low (Fig. 1). Generally speaking, for each product considered separately, the higher its N dosage, the higher its N uptake and yield (Fig. 1). The N uptake was, on average, higher with the N treatments than without (PK_CL and UNF_CL) (Table S2). All five of the high-dosage N treatments were statistically superior ($p < 0.01$) to the controls (Table S4), but not the medium and low dosages. The only exception to the latter was the highly significant contrast ($p < 0.01$) between PS_high and PS_low (Fig. 1 and Table S4). Inter alia, when comparing the BBF treatments with the CAN benchmarks, no significant differences were noted between the BBFs and the CAN references at all dosages (Table S4).

The NUE of CAN treatments were in a similar range (27–34 %). The NUE of PS_low was strikingly low with 5 ± 18 %, however caution must be exerted in light of the high standard deviation. As with the previous years, the general lack of salient differences in dosage-response affected the interpretation of NUE and NFRV metrics. Effect size statistics indicated that, in comparison with the CAN_high benchmark, PS_high stood out as having a 95 and 97 % probability of being non-inferior in terms of dry yield and N uptake, respectively (Table S8 and Fig. S6 **C**). The PU_high was also slightly above the CAN_high reference which translated to a 37 % probability of being non-inferior. This being said, as a point of reference, the average yield of early potatoes in the Flemish region in 2021 was 40.3 t ha^{-1} (Belgian statistical office (Statbel), 2023) against 20.6 t ha^{-1} of fresh tubers on average in this trial (excluding control treatments), in other words under half (49 %) the average regional yield. Hence, suboptimal yields were observed for all three crops (discussed hereafter).

3.2. Environmental impact: residual mineral N of the soil profile (0–90 cm) at the end of the Flemish legal period

3.2.1. Maize (year 1)

Although there were no significant contrasts, the residual nitrates (0–90 cm) tended to be lower with the two control treatments than with the N treatments (Fig. 1). Concentration of NO_3^-–N was generally higher in the 30–60 cm and 60–90 cm layers than at 0–30 cm (Fig. 2). At 30–60 cm, the residual NO_3^-–N from the CAN_high treatment was higher than PK_CL and UNF_CL. Likewise, CAN_low was also higher than PK_CL and UNF_CL. Regarding effect size statistics, the BBFs with the highest probability of residual NO_3^-–N levels that would not be in excess of the CAN_high benchmark average were as follows: LFD_low (65 %), LFD_med (54 %), PS_med (48 %), PS_low (46 %), PU_high (39 %) (Table S6). In this respect, these BBFs performed better than CAN_high. As alluded to previously, for year 1 in particular, in many instances the amounts of N that were actually applied were either undershot, by 30 % in the case of PS treatments, or overshot by close to 30–40 % in the case of the D treatments, many BBFs lying somewhere in between (Table 3). Nevertheless, these discrepancies did not lead to statistically meaningful differences between treatments.

To comply with the European Nitrates Directive (91/676/EEC), the Flemish government has imposed ceilings (depending on the zone, soil type, crop) for nitrates in the soil profile up to 90 cm deep. This assessment is carried out during the wintertime — between October 1st and November 15th — as these residual nitrates are considered as the main determinant of nitrate leaching to surface and groundwater (Vandendriessche et al., 2011). Harvest (end of September) was immediately followed by heavy outbursts of rain which delayed soil sampling for residual nitrates for more than a fortnight (until mid–October). In this short amount of time, the local weather station on the nearby farm recorded 106 mm of rain. Consequently, a substantial leaching of nitrates likely took place in this short yet critical timeframe, especially considering the light and sandy soil texture. This assumption was supported by the observation of the nitrate distribution between the layers of soil post-harvest, where the NO_3^-–N had migrated from the topsoil, with 16 kg NO_3^-–N ha^{-1} on average, to the deeper layers, with 38 and 35 kg NO_3^-–N ha^{-1} in the 30–60 cm and 60–90 cm layers, respectively (Fig. 2). The legal threshold in Flanders for maize on sandy crops is 80 kg NO_3^-–N ha^{-1} meaning that most N treatments were above the mark (Fig. 1). The exceptions were LFD_low (73 kg NO_3^-–N ha^{-1}), LFD_med (77 kg NO_3^-–N ha^{-1}), PS_low (79 kg NO_3^-–N ha^{-1}) and PS_med (79.7 kg NO_3^-–N ha^{-1}), thus most barely qualified.

3.2.2. Spinach (year 2)

As a general pattern, NO_3^-–N tended to increase from the topsoil downwards (Fig. 2). The dosage effect was on the edge of significance ($p = 0.0751$, Table S1). The post-hoc analysis returned a significant difference ($p < 0.05$) between PU_med and UNF_CL in reason of the former showcasing an exceptionally higher value comparatively to the other treatments, yet the high standard deviation associated with PU_med calls for circumspection as mentioned previously (Fig. 1). Regarding the non-superiority test, the BBFs with the highest probability of residual NO_3^-–N levels that would not be in excess of the CAN_high benchmark average were as follows: LFD_med (42 %), PU_low (26 %), D_low (20 %), LFD_low (16 %), PS_low (12 %) (Table S7 and Fig. S6 **B**).

For each of the soil depths considered separately, no significant differences in NH_4^+–N ($p > 0.05$) were found. For NO_3^-–N, there were only a handful of significant contrasts that did not bare any fundamental implications on the general trend. Namely, at the 0–30 cm depth,

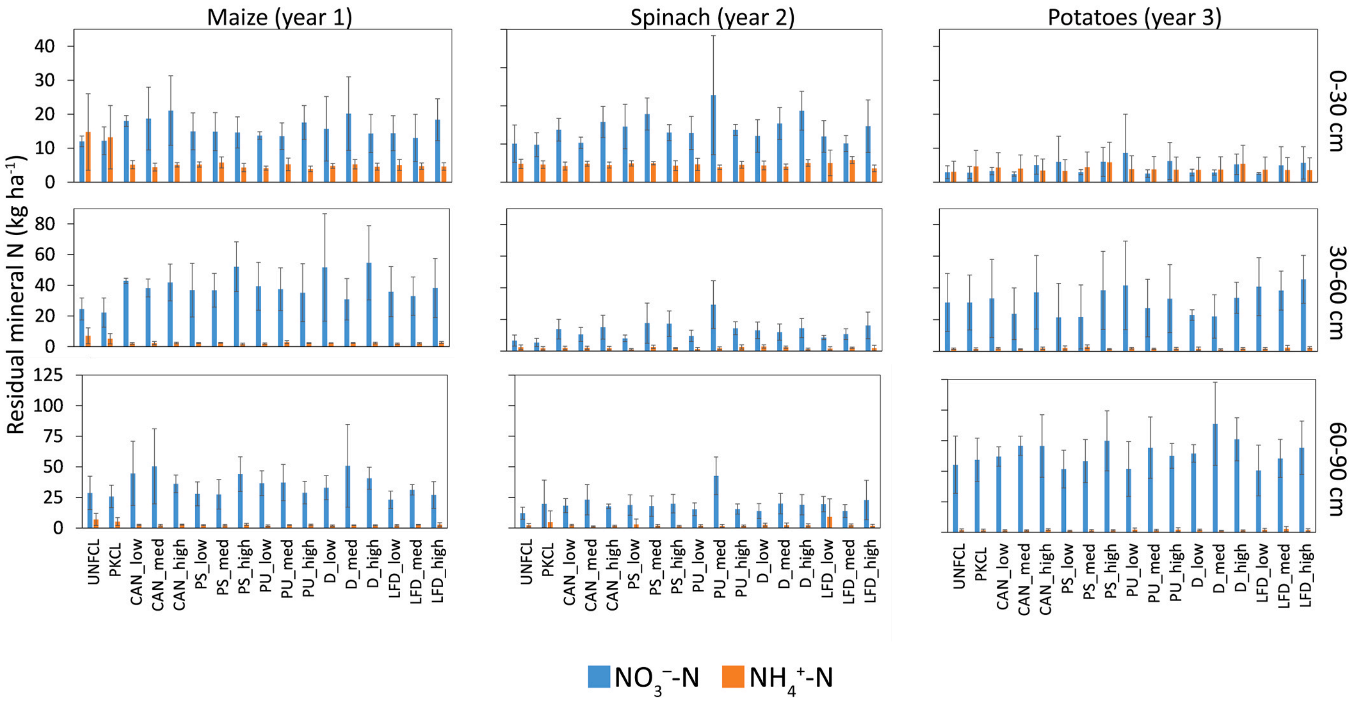

Fig. 2. Distribution of soil residual mineral N (NH_4^+–N and NO_3^-–N) at three different depths (0–30; 30–60; 60–90 cm) as detected post–harvest at the end of the Flemish legal period.

CAN_high (16 kg NO_3^-–N ha^{-1}) was significantly higher ($p = 0.0449$) than CAN_med (10 kg NO_3^-–N ha^{-1}). At 30–60 cm, NO_3^-–N from PU_med was significantly different from PK_CL ($p = 0.0119$) and UNF_CL ($p = 0.0202$). At 60–90 cm, PU_med (28 kg NO_3^-–N ha^{-1}) was significantly higher than D_low (14 kg NO_3^-–N ha^{-1}) and UNF_CL (12 kg NO_3^-–N ha^{-1}) ($p = 0.0173$ and $p = 0.0018$, respectively). Hence, apart from the cases specified above, not even the controls (UNF_CL and PK_CL) were any different statistically than the N treatments at any given soil depth. An exceptionally high value of 43 kg NO_3^-–N ha^{-1} was observed at 60–90 cm depth from PU_med, which naturally carried over to the NO_3^-–N concentration contained in the entire soil profile (Fig. 1).

The year 2020 was dry in comparison with the thirty–year reference period (Fig. S5). Overall, the season was characterised by an uneven distribution of rainfall with exceptionally wet months interspersed with unusually dry periods (Fig. S5). The soil sampled before the trial for characterisation of nitrates was done so during a relatively wet month of February. At that time, nitrates were low (7 kg NO_3^-–N ha^{-1} on average) in the upper layer (0–30 cm) and increased gradually with depth in the 30–60 cm layer (13 kg NO_3^-–N ha^{-1} on average) and 60–90 cm (18 kg NO_3^-–N ha^{-1} on average) (Fig. S7), probably corresponding to the remaining mineral N that leached into the deeper layers at the end of the winter, and which in all likelihood would not have been accessible to the spinach's shallow root system this early on (Frerichs et al., 2022). The fertilisation schemes were applied at the end of March, and the seeds were sown shortly after, in mid–April (Table 1). Precipitation in February and March was particularly heavy, with 68 and 58 mm of rainfall, respectively, above the reference values for those months (Fig. S5).

One day after harvest, soil sampling for analysis of residual nitrates and ammonium was carried out. There was a high variability between treatments, but the largest part of the nitrates was still concentrated in the topsoil (0–30 cm) with 36 kg NO_3^-–N ha^{-1} on average. Results were quite scattered as for instance PU_low had 18 kg NO_3^-–N ha^{-1} while on the other extreme CAN_low was the highest with 63 kg NO_3^-–N ha^{-1} (Fig. S7). The concentration of ammonium in the topsoil was still quite high with an average 9 kg NH_4^+–N ha^{-1} (Fig. S7). This could be due to the dry conditions which were not conducive to optimal infiltration of the fertilisers in the soil. Also, the relatively high concentrations of NH_4^+–N in the topsoil (two months after initial application) might also suggest that the microbial nitrification activity was impaired by a lack of moisture. In turn, ammonia volatilisation would have been considerable for some BBFs. Furthermore, the effects of the water-and-heat stress on the plant's physiology likely reduced the uptake of nutrients, resulting in higher mineral N levels in the topsoil. The topsoil showed unusually high EC values, including the control treatments. Whereas for maize and potatoes the average EC was 46 ± 7 mS cm^{-1} and 66 ± 15 mS cm^{-1}, respectively, it was 132 ± 39 mS cm^{-1} for the spinach trial. Hence the high EC, measured at the tail end of a very dry growing season (immediately after harvest), could signal that evapotranspiration was such that it brought about an accumulation of salts in the topsoil via capillary rise.

The residual nitrates determined in the soil at the end of the legal period in Flanders (end of October) were considerably lower than those that had been measured immediately after harvest. In the topsoil, nitrates were on average 10 kg NO_3^-–N ha^{-1} (Fig. S7) with a similar concentration found in the middle layer (30–60 cm). The bottom layer (60–90 cm) was more nitrate-heavy (19 kg NO_3^-–N ha^{-1} on average) indicating that some leaching had occurred in reason of the high post-harvest precipitations. In effect, shortly after harvest, the month of June was exceptionally wet (+71 mm), and the months of July through October delivered more rainfall despite being drier than the reference period (Fig. S5). An intermediary crop of maize, established after the spinach, was harvested end of September (Table 1). While its N uptake was not measured, its presence likely contributed to limiting the leaching of nitrates in the two combined upper layers (0–60 cm). As a result, the treatments mostly complied with the Flemish legal limit of 80 kg NO_3^-–N ha^{-1}, the only exception was PU_med (95 NO_3^-–N ha^{-1}) which had a very high variability (discussed previously).

3.2.3. *Potatoes (year 3)*

The post-hoc test did not reveal any significant contrasts between treatments (Table S5). The non-superiority test (Fig. S6 **C**) showed the BBFs with the highest probability of residual NO_3^-–N levels that would not be in excess of the CAN_high benchmark average to be PS_low (80 %), PS_med (72 %), D_low (52 %), LFD_low (41 %), PU_med (26 %), PU_high (18 %), PU_low (18 %) (Table S8). Thus, these BBFs performed better than the CAN_high benchmark (15 % tolerance) and in general, year 3 seemed to have produced the overall best performance in this regard. This trend would partly confirm previous findings reporting a higher propensity for nitrate leaching from mineral N treatments than digestate (Tsachidou et al., 2019).

There were no significant differences ($p > 0.05$), either in NH_4^+–N or NO_3^-–N, at any of the three depths considered individually, once more highlighting the absence of any significant impact of the fertilisers or dosages. Statistical comparisons aside, for all treatments (including controls), there was still a perceptible trend of increasing NO_3^-–N with soil depth, or in other words there was less NO_3^-–N in the topsoil (0–30 cm) than in the middle layer (30–60 cm) than in the deepest layer (60–90 cm) (Fig. 2). There was a minor presence of NH_4^+–N, which was slightly higher in the topsoil layer (0–30 cm) — 4 kg NH_4^+–N ha^{-1} on average — than the two deeper layers (2 kg NH_4^+–N ha^{-1} each) (Fig. 2) while these values for NH_4^+–N were consistent, across the 3 years, with reported ranges below 10 kg NH_4^+–N ha^{-1} (Tsachidou et al., 2019).

The total annual precipitations were below those of the thirty–year reference rainfall index (Fig. S5). A wetter-than-average month of January (+25 mm) and drier month of February (–37 mm) roughly evened out the balance at the moment of the sampling of residual mineral N on the first day of March, prior to sowing (Table 1). On all plots, the mineral N was low, between 3 and 4 kg NO_3^-–N ha^{-1} on average in the 0–30 and 30–60 cm layers (Fig. S7). The NH_4^+–N was about half that value at all three depths (2 kg NH_4^+–N ha^{-1} on average). A moderately higher level of NO_3^-–N of 9 kg NO_3^-–N ha^{-1} was present in the 60–90 cm layer, indicating that some leaching from the previous spinach crop had taken place over the winter. As the nitrates were around 20 kg NO_3^-–N ha^{-1} (0–90 cm) at the end of October of the previous year (Section 3.2.2), it can be assumed that about half this amount percolated beyond reach to the deeper horizons.

The month of May was unusually wet with 90 mm of rainfall and 32 mm above seasonal average (Fig. S5). While the month of June was also slightly wetter than average, the period from July to September was drier than usual with 36, 20, 43 mm less, respectively (Fig. S5). The abundant rainfall in May and June, followed by moderate precipitation up until the harvest at the beginning of August, and then again heavier rainfall in October and November (Fig. S5), seemed to have been propitious for both nitrification activity and leaching based on two observations. Firstly, indicative of a strong nitrifying activity, the presence of ammonia measured in November was almost negligible, with 4 kg NH_4^+–N ha^{-1} in the topsoil and 2 kg NH_4^+–N ha^{-1} each at 30–60 and 60–90 cm. Secondly, the residual nitrates in the topsoil were now at around 4 kg NO_3^-–N ha^{-1} on average, meaning they subsided considerably while, at the same time, the 30–60 cm depth contained roughly the same amount as was detected during the August harvest (32 kg NO_3^-–N ha^{-1} on average), but more strikingly the 60–90 cm layer now held 65 kg NO_3^-–N ha^{-1} on average. Hence, the effects of the heavy rainfall on leaching into the deeper layer could be observed as the quantities of nitrates that were present in all three layers at harvest (August) could be found in almost identical amounts in November, only the nitrates had now increasingly percolated to the deeper layers (Fig. S7). As a concomitant factor, the ongoing soil and fertiliser mineralisation post–harvest assuredly also played a part in the cumulated winter leaching.

Potato crops are generally prone to nitrate leaching (Venterea et al., 2011). Application of N early in the season is recommended to ensure an optimal vegetative growth which translates to higher yields down the road (Stark et al., 2020), but it also tends to increase the risk of nitrates leaching especially on sandy soils (Clément et al., 2021; Kelling et al., 2015) as the N uptake increases over time and only reaches its peak in the last stage of tuber bulking, shortly before harvest (Jia et al., 2018). The ryegrass installed shortly after harvesting the potatoes might have helped in reducing leaching in the topsoil (0–30 cm) and even out the differences between fertilised and unfertilised plots, but its overall effect was visibly insufficient. In fact, the Flemish legal limit for residual nitrates measured in the wintertime for potatoes is set at 90 kg NO_3^-–N ha^{-1} while the majority of the N treatments were above this legal threshold (Fig. 1). The only exceptions were PS_low and PS_med (79 and 83 kg NO_3^-–N ha^{-1}, respectively), while D_low was, strictly speaking, above the limit (90.3 kg NO_3^-–N ha^{-1}).

3.3. *Effect of pedoclimatic conditions on nitrates distribution and agronomic performance*

On top of a warmer climate (between +2.6 and +3.5°C), the latest Climate Adaptation Plan from the Belgian National Climate Commission expects increased seasonality of precipitations and more extreme events such as thunderstorms, heatwaves, or heavy rains by 2100 (NCC, 2016). In many regards, the present field trial already provided a glimpse of things to come. The unfavourable weather conditions — unequal distribution of rainfall, extreme heat events and at times drought — had a determining impact on the agronomic and environmental metrics of the present study across the 3 years. It resulted in widely distributed data which irremediably affected the statistical significance of the outcome, greatly weighing down on the ability to parse signal from noise, whereas a narrower data variability could have brought to light a more clear-cut statistical significance between treatments. Regarding the effect of weather, a similar study was conducted over three years in the exact same field (2011–2013), which combined synthetic fertiliser and either the liquid fraction of digestate or pig slurry, with amounts of total N varying between 157 and 305 kg N ha^{-1}. The first year, residual nitrates from all treatments were above the 80 kg NO_3^-–N ha^{-1} limit (even pushing beyond 100 kg NO_3^-–N ha^{-1}) (Sigurnjak et al., 2017). As in the present study, residual nitrates in the 0–90 cm soil layer varied greatly from one year to the other, almost by a factor of 2. Unlike the present study, as maize was grown three years in a row, it was found that the effect of the experimental year ($p < 0.05$) was stronger than the effect of the treatments (Sigurnjak et al., 2017), which echoes our present findings.

3.4. *Nitrogen stewardship*

3.4.1. *Synchronisation between nutrient characterisation of BBFs and time of application*

It became apparent during this study that the N content of BBFs could vary sometimes considerably in a relatively short timeframe. During the first year, regional regulations pertaining to the transport of manure in Flanders made it difficult to store the BBFs onsite ahead of time. For this reason, there was a four–week window between the first series of sampling (which had to be done at each of the respective farms where the BBFs were being produced) and the second sampling for nutrient characterisation on the day of the trial. In this timeframe, the N content of the BBFs had varied anywhere between –33 % to +37 %, much of it owing to the heterogeneity between the products that were sampled once from the slurry pits and the subsequent samples that were transported to the field. The administrative hurdle having been sorted out by year 2, the storage of the BBFs onsite earlier in the year was made possible, thus samples could be taken in the days leading to the trial to cross-check the nutrient composition. As a result of both sampling points being closer to the time of application and also coming from the same batch, the accuracy of the N characterisation for years 2 and 3 were dramatically improved. In spite of this, there were still some discrepancies between N targets and N applied (5 % for D treatments in year 2; 14 % for PS in year 3). This shows that BBFs should be sampled, at least twice, as close as possible to the moment they will be utilised in the field (one of the samples should be on the same day to account for slight variations). Thus, the timely monitoring of the nutrient composition and the thorough mixing of the BBFs have a significant impact on the accuracy of the calculated BBF application rates and constitute important steps to curb any over-or-under-fertilisation. Given the possibility, the somewhat time-consuming wet characterisations could be bypassed with mobile nuclear magnetic resonance sensors, which allow for the online monitoring of nutrients (Sørensen et al., 2015), or near infrared spectroscopy (Gogé et al., 2021), both of which provide accurate data for crucial elements such as N, P and K. Such technologies offer the advantage of delivering the analyses almost instantaneously which can greatly facilitate the critical decision-making process and reactivity as regards the exact amounts of BBF to be applied.

3.4.2. Soil characteristics and the use of catch crops

Aside from weather conditions, the sandy soil in this field also played a part in the N dynamics, as the coarser texture and lower water-holding capacity would have been highly conducive to nitrates leaching (Gaines and Gaines, 1994). Moreover, for the spinach (year 2), the N from the BBF had to be delivered to the root zone once the crop had been sown. This meant that conventional ploughing was not an option, as the fertilisers would have been buried too deep (20 cm) in relation to the roots. Instead, non-reversing soil tillage was opted for, but with this technique, any crop residues from previous catch crops would have resulted in a coarse seedbed which can detrimentally affect plant emergence and growth. As a result of these technical constraints, in year 1, the soil was left bare over the winter, while it has been extensively reported that nitrates leaching can be reduced significantly through the use of catch crops (Hansen et al., 2007; Lewan, 1994).

During the first year, the abundant rain on a dry and warm soil, between the moment of harvest and the determination of residual nitrates, greatly favoured N leaching and mineralisation. In year 2, maize was sown immediately after the spinach and harvested prior to the determination of the residual nitrates in the winter. It stands to reason that this intermediary crop (not assessed) was beneficial in reducing residual nitrates that year. An additional explanation would lie in the fact that, at harvest, the spinach was cut at 6 cm aboveground, thus a sizeable amount of N would still have been locked up in the crop residues and roots. Due to a high variability in the data, only a handful of the higher dosage treatments were significantly different from the controls. However, if we consider the averages at face value, as an N-demanding crop, it appears the N regimes had an effect (although not statistically speaking) on the N uptake and yields of spinach (Fig. 1). At the end of year 3, as with year 1, residual nitrates were mostly above the legal threshold. The ryegrass seemed to have been insufficient in this instance, probably in reason of the heavy rain on a sandy soil in the months leading to the sampling of residual nitrates in November.

3.4.3. Long-term effect of organic nitrogen from BBFs

Another point deserving of attention in the future is the long-term effect of the organic N such BBFs bring to the field year in, year out. In year 3, the fact that the control plots, having received no N, ranged between 89 and 93 kg NO_3^-–N ha^{-1} at the end of the season drew our attention. Similarly, in year 1, the UNF_CL contained 65 kg NO_3^-–N ha^{-1} and 29 kg NH_4^+–N ha^{-1} (Fig. 2), hence while it did not exceed the 80 kg NO_3^-–N ha^{-1} legal threshold for maize crop at the time of sampling, it can be hypothesised that the ongoing nitrification had the potential to tip the scales beyond the legal limit. In this particular field, before the onset of the study, it was customary to apply 50 t manure ha^{-1} y^{-1}. Based on the initial characterisation of the soil before the trial (Section 2.1.1), it contained about 2.64 t organic N ha^{-1}. Thus, a possible explanation for the mineral-N rich control plots might be found in the repeated application of organic fertilisers (manure). This could have prompted a buildup of organically bound N in the soil over time and a slow mineralisation over the long-term (Gutser et al., 2005; Sogn et al., 2018; Tsachidou et al., 2019) — the dynamics of which are more difficult to predict (Schröder et al., 2013) — and the effects of which might have been underestimated in this study. After all, Flanders has historically been home to most of the intensive livestock production in Belgium, which over time led to a manure surplus, accompanied by various strategies (on top of the Nitrates Directive) to limit the resulting regional nutrient overload (manure exportation, denitrification, anaerobic digestion) (Coppens et al., 2016). In spite of these efforts, many years of intensive mineral fertilisation and liberal spreading of manure caused Flemish agricultural soils to have high to very high chemical fertility (N and P in particular) (De Neve et al., 2006). The residual nitrates measured over the course of this study tended to substantiate the claim of an initially nutrient-rich soil. It would then stand to reason that prudent measures might be envisaged to encourage the exhaustion of the current nutrient pool, by observing a more conservative fertilisation scheme for a short period of time for example, before resuming a more balanced crop fertilisation strategy.

3.4.4. Ammonia volatilisation in relation to the fertiliser application strategy

The effect of tillage on N dynamics was not examined: for years 1 and 3, the fertilisers were ploughed to a depth of 20 cm before planting; in year 2, non-reversing soil tillage was carried out as a result of which the fertilisers remained on the surface. Assumedly, the custom-designed injection system (Section 2.1.3) significantly reduced ammonia volatilisation. As an order of magnitude, an open field trial reported that digestate injected at 15 cm deep resulted in a 12 % volatilisation of total ammonia nitrogen (Zilio et al., 2021). Another study, comparing broadcasting and shallow injection of digestate reported that NH_3 emissions dropped by up to 50 % with the latter (Nicholson et al., 2018). Hence, especially in the scenario of surface application in year 2, the intensity of the losses by volatilisation remains unknown. Although this metric was not the focus of this study, such losses could have been sizeable enough that the NUE values may have been misleading. It has already been suggested that, as valuable as the NUE metric can be in some cases, it provides an incomplete picture at best as it is influenced by several unaccounted factors (weather, soil texture, N losses) (Sigurnjak et al., 2017). Moreover, the fluctuation in soil mineral N in a single growing season can be considerable. In the simplest of terms, the synchrony between crop N uptake (demand) and soil available N (offer) is not accurately reflected in most NUE indices as, even if a substantial amount of soil available N is measured at the beginning of the growing season, this does not guarantee that sufficient amounts will be available when the crop most needs it. Hence, as alluded to above, the portion of N that was lost (via leaching, volatilisation, denitrification, immobilisation) by the time the plant was actively absorbing N would not be accurately accounted for by the NUE (Congreves et al., 2021). For instance, in year 3, the particularly wet months of May and June led to heavy leaching to the deeper layers, which opens up the possibility that the plant's N uptake might not have been optimal — final yields were after all quite low — and that, even at the higher N regimes, the crop might have paradoxically been N-limited as a result of a poor temporal overlap between N supply and plant demand. This is why the inclusion of other indicators (root N pools, plant N synchrony, N forms other than mineral, a more precise fate of N via N–15 labelling) could improve the overall accuracy of the NUE index (Congreves et al., 2021).

3.4.5. Single or split application of fertilisers

Nowadays, the current NUE values for most crops worldwide are estimated to be no higher than 50 % at best (Fageria and Baligar, 2005). This estimation agrees with a more recent study which places this value at 44 % globally (Chatzimpiros and Harchaoui, 2023). The NUE values in the present study were for the most part in line with this range (Table S2). As indicated by those authors, these relatively low NUE values mean there is ample room to improve N recovery by the plant and highlights the need for more integrated N management strategies (some of which were already discussed above). One such strategy consists in split-applying N over the course of the growing season. The general principle is that only a fraction of the total N dose (30–50 %) is applied at sowing, the remainder is applied later in the growing season (two–-way or three–way split), if deemed necessary, to allow for a better spatial and temporal adequacy between the plant's needs and the soil available N supplied by the fertilisers (Riar and Coventry, 2012). Several studies reported this strategy to have increased yields (Coventry et al., 2011) and reduced pollution by nitrates (Lu et al., 2021), resulting in an overall improved NUE (Souza et al., 2020). However, the empirical results reported in literature are seldom generalisable and site specific strategies should always be preferred (Riar and Coventry, 2012).

Incidentally, split fertilisation was picked up by Flemish authorities and recommended as a good agricultural practice as part of the Flemish Manure Action Programme. For potatoes in particular, which are drought-sensitive and require a high N fertilisation yet have a low N uptake, the potential for reducing nitrate losses with split-N strategies is considered to be high in Flanders (Nawara et al., 2021). Without readdressing the strong influence, discussed above, of the pedoclimatic conditions on the fixed effects and variables that were the object of this study, broadly, it appears the crop N needs did not coincide optimally with the soil available N supplied by the BBFs and the synthetic benchmark alike. Indeed, in two of the three years, soil residual nitrates (0–90 cm) were predominantly above the allowed limit — even with some of the lower N regimes — while yields were generally low to moderate for the three crops. The protocol that was observed during this trial was to deliver the fertiliser N as a single dose at the beginning of each growing season. This raises the question, probably worth investigating in the future, of whether a split-application of N fertilisers would not have been more efficient for increasing N uptake and reducing pollution by nitrates. In addition, increasing the frequency of the samplings would provide a higher resolution and better understanding of the nitrate dynamics. Lastly, augmenting the number of replicates for such field experiments would have gone a long way in increasing the statistical power and interpretation of results.

The ANOVA tests did not reveal any meaningful differences, either in agronomic performance (crop yield and N uptake) or environmental impact (residual nitrates) between the BBFs and the synthetic fertilisers. This being said, the non-superiority tests (95 % confidence interval) on residual nitrates comparing BBFs with the CAN_high treatment, taken as reference, showed a general tendency of BBFs to perform better, although results varied greatly from year-to-year. Of course, the high temporal and spatial heterogeneity of the field conditions, and weather events, must be taken into consideration in the interpretation of our results. Nonetheless, the current findings were in line with other studies that reported similar performances between digestates and synthetic treatments as short-term fertilisers with a fast mineral N release capacity (Grillo et al., 2021; Luo et al., 2022; Tsachidou et al., 2019).

4. Conclusion

Over the course of the 3–year field trial, yields were low to moderate which suggests the unfavourable weather conditions in combination with the soil type (sandy) had a significant impact on the outcome of the study. The combined effects of uneven seasonal distribution of precipitations, periods of drought, interspersed with heavy episodes of rainfall interfered with the expected dose-response of the three incremental N dosages. In spite of this, in years 2 and 3, a meaningful dosage effect ($p < 0.05$) on yield and N uptake was observed between some of the higher fertiliser regimes (BBFs and synthetic) on one hand, and the unfertilised control treatments on the other. No significant differences ($p > 0.05$) were observed between the BBFs and the synthetic fertiliser benchmarks in terms of agronomic performance (defined as crop N uptake and crop yield). There were no significant differences ($p > 0.05$) in soil residual nitrates (0–90 cm) between BBFs and synthetic fertilisers — irrespective of the dosage applied — neither were there any differences compared with the unfertilised controls, pointing to the presence of an already N-rich soil (before fertilisation) that further encroached on the dosage effect.

However, the non-superiority test underlined a general tendency of BBFs to perform better than the CAN_high reference, despite results varying from year-to-year. A few treatments aside, residual nitrates from most treatments (synthetic and BBF) were above the legal limit two years out of the three, probably the consequence of rapid rain bursts on a sandy N-rich soil. This fact underpins a propensity for nitrate leaching from BBFs and synthetic fertilisers alike and raises the more general question of the observance of integrated N management strategies to increase overall N efficiency and cut back on N losses to the environment. In our view, another point deserving of attention is the long-term effect of such BBFs on soil properties. In particular, the regular application of BBFs can convey sizeable amounts of organic N to the soil, whose long-term contribution to N mineralisation, and potentially unchecked leaching, needs to be further elucidated.

Lastly, during the storage of the BBFs before field application, it became apparent that their mineral N content was subject to change over time. As a consequence, nutrient determination should be carried out at least twice, and as close as possible to the day of application, as these steps significantly increased the accuracy of N accountancy.

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CRediT authorship contribution statement

Ivona Sigurnjak: Conceptualization, Methodology, Writing – review & editing. **Harmen Dekker:** Funding acquisition, Project administration, Resources. **Erik Meers:** Conceptualization, Funding acquisition, Project administration, Resources, Writing – review & editing. **Tomas Van de Sande:** Conceptualization, Data curation, Formal analysis, Investigation, Methodology, Writing – review & editing. **Gregory Reuland:** Data curation, Formal analysis, Investigation, Validation, Writing – original draft, Writing – review & editing.

Declaration of Competing Interest

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

Data availability

Data will be made available on request.

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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.eja.2024.127380.

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