

RESEARCH ARTICLE

Sewage sludge showed high agronomic value, releasing nitrogen faster than farmyard manure

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Abstract

The use of fertilizers in agriculture, particularly organic fertilizers such as sewage sludge, is a key research priority due to its impacts on crop productivity, production costs, environmental risks and the push for a more circular economy. This study, conducted in Bragança, northeastern Portugal, focused on forage maize during the 2022 and 2023 growing seasons. Eight fertilization treatments were established corresponding to the application of mineral nitrogen (N) at rates of 50 (N50), 100 (N100), 150 (N150) and 200 (N200) kg ha⁻¹, three organic amendments applied at a rate equivalent to 200 kg ha⁻¹ of N, namely sewage sludge (SS200), cow manure (CM200) and sheep manure (SM200), and a non-fertilized control (N0), with the aim of assessing the agronomic value of these treatments for forage maize (*Zea mays* L.). Maize dry matter yield (DMY) ranged from 10.8 to 20.3 t ha⁻¹ in 2022 and 13.7 to 23.6 t ha⁻¹ in 2023 for N0 and N200, respectively. Organic amendments produced 14.7–17.7 t ha⁻¹ in 2022 and 20.5–24.4 t ha⁻¹ in 2023. Increased mineral N rates resulted in higher soil inorganic N content, N concentration in leaves and N recovery in tissues, with organic amendments showing lower values than crops fertilized with N200. However, due to cumulative nutrient release, organic amendments improved DMY and N use efficiency in the second year. Sewage sludge mineralized rapidly due to its low carbon (C)/N ratio and absence of hard-to-degrade compounds. It is also rich in phosphorus (P), enhancing its fertilizing value. Although nitrate leaching and denitrification potential appeared lower with organic amendments, long-term applications may increase risks, requiring careful monitoring to ensure sustainable and safe practices.

KEYWORDS

circular economy, maize yield, nitrogen use efficiency, organic fertilization, soil inorganic nitrogen, *Zea mays*

1 | INTRODUCTION

Soils have the potential to contain considerable quantities of N, largely determined by their organic matter content. However, the availability of mineral N in the soil solution or adsorbed to the exchangeable complex is

generally low, typically ranging from 1% to 2% of the total N content in the soil (Weil & Brady, 2017). Consequently, crops heavily rely on the regular application of N fertilizers, significantly increasing their use in agriculture (Havlin et al., 2017). Globally, agricultural N fertilizer usage has surged over the past 50 years, escalating from

36.1 Mt. in 1972 to 108.1 Mt. in 2022 (FAOSTAT, 2024), with an annual growth rate of 2%. This increase can be attributed to the continuous intensification of farming practices and the well-established positive impact of N application on crop yield. Nitrogen fertilizers improve plant N nutritional status, photosynthetic efficiency and various morphological traits such as plant height, branching and leaf area index, collectively increasing total DMY, grain yield or fruit yield (Deng et al., 2023; Pasley et al., 2020; Patanita et al., 2019).

While the positive effects of N on crop productivity are widely recognized, it is essential to acknowledge the potential negative environmental consequences as well. Over 50% of N applied as fertilizer to agricultural fields is lost to the environment, mainly as nitrate-N to watercourses and N oxides (NO_x) to the atmosphere (Roy, 2022). The environmental impacts encompass diverse issues such as water eutrophication, soil salinization, atmospheric contamination with greenhouse gases and reduction of ecosystem biodiversity (Marques et al., 2022; Roy, 2022; Weil & Brady, 2017). Moreover, N fertilizers are synthesized through the energy-intensive Haber-Bosch process, requiring high pressure and temperature to break the stable triple bond of dinitrogen (N₂) molecules. This process consumes 28 GJ t⁻¹ per unit of synthesized ammonia (NH₃), with industrial N production accounting for 1% of global energy consumption. Notably, 1.9t of carbon dioxide (CO₂) is emitted per t of NH₃ produced, potentially hindering efforts toward C neutrality in many countries (Basu & Ganguly, 2023; Petukhov et al., 2022). Hence, the global concern regarding climate change and the environmental repercussions of excessive use of conventional N fertilizers fosters a renewed effort to explore alternative fertilization strategies.

Organic amendments can potentially increase crop productivity (Arrobas et al., 2022; Dimande et al., 2023; Luo et al., 2018; Rodrigues et al., 2018). Generally, the release of nutrients from organic amendments occurs slowly and progressively as the organic substrate mineralizes, reducing the risk of excessive accumulation of mineral N in the soil, which can lead to its loss (Myrold & Bottomley, 2008; Weil & Brady, 2017). Furthermore, a meta-analysis based on long-term experiments in Europe highlighted an additional positive effect on crops from organic inputs, even in cases where macronutrients were not limiting factors (Hijbeek et al., 2017). The role of organic amendments in increasing soil organic matter and enhancing its physical, chemical and biological properties (Cardarelli et al., 2023; Liu et al., 2022; Wang et al., 2021) may explain the observed increase in crop productivity, which extends beyond the simple effect of nutrient release. This phenomenon, often described as the 'manuring effect' (Dimande et al., 2023; Rodrigues et al., 2018; Weil & Brady, 2017), highlights the

distinct advantages of organic amendments. Accordingly, the application of organic amendments can enhance crop productivity in ways that cannot be fully achieved using mineral fertilizers alone.

Agricultural and livestock activities have become increasingly separated due to the specialization of their operations, making farmyard manure not readily available to all farmers. Consequently, the use of alternative organic resources has become necessary. Sewage sludge emerges as a nutrient-rich and increasingly accessible resource with significant potential for agricultural use (Barros et al., 2023; Eid et al., 2022; Tsadilas et al., 2018). However, studies demonstrating its agronomic value in promoting plant growth are limited, and producers still lack sufficient guidelines for its effective and informed application.

Thus, this study aims to evaluate the agronomic value of sewage sludge by assessing its impact on soil nutrient availability, the nutritional status of silage maize, and DMY, in comparison to two commonly used organic amendments in the region: cattle manure and sheep manure. The experimental design also includes varying rates of mineral N fertilizer to create an N response curve and assess the potential of organic amendments to replace mineral N fertilizers. Consequently, the study hypothesizes that (i) sewage sludge possesses a fertilizer value equivalent to animal manure and (ii) organic amendments can fully substitute mineral N fertilization.

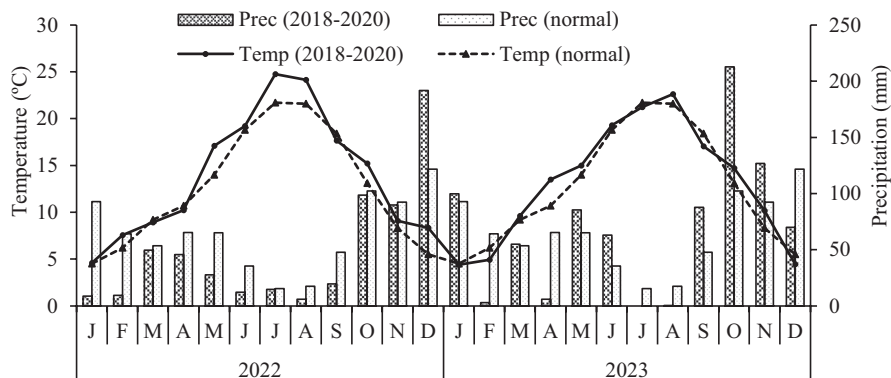
2 | MATERIALS AND METHODS

2.1 | General characterization of the study site

The field experiment was carried out between 2022 and 2023 at Quinta de Santa Apolónia (41°46'50.2"N, 6°47'53.4"W) in Bragança, northeastern Portugal. Maize (hybrid DKC 6181, mid-season FAO 500) was grown for silage during the summer months, between May and September. Maize cultivation is part of an eight-year irrigation rotation, which includes a four-year ley phase with pasture seeded with a mixture of legumes and grasses and grazed by a herd of cows. The following 4 years consist of a monoculture phase of maize for silage. This study occurred during maize cultivation's third and fourth years, representing the rotation cycle's final 2 years.

The region benefits from a Mediterranean climate with some Atlantic influence, classified as Csb, corresponding to a temperate climate with dry and mild summers (IPMA, 2024). The average annual temperature is 12.6°C, and the annual precipitation is 772.7 mm (IPMA, 2024).

FIGURE 1 Monthly mean air temperature and precipitation values from the climatological normal of the region (1981–2010) and during the period of the field experiments.



Monthly values of the 1981–2010 climatological normal and observed mean air temperature and cumulative precipitation during the experimental period are presented in Figure 1.

The soil where the study was carried out is classified as eutric Fluvisol (WRB, 2015) with a sandy clay loamy texture (540, 250, and 210 g kg⁻¹ sand, silt and clay, respectively). Some of the soil properties determined at the beginning of the experiment, based on composite soil samples collected in a zigzag pattern throughout the experimental plot from the 0–0.20 m layer, were as follows: pH (H₂O) 6.2; organic C 19.0 g kg⁻¹; extractable P (as P₂O₅) and potassium (K, as K₂O) (determined by the Egnér-Riehm method) 109.6 and 101.8 mg kg⁻¹, respectively; extractable boron (B) 2.2 mg kg⁻¹; exchangeable calcium (Ca), magnesium (Mg), K, and sodium (Na) 13.7, 7.1, 0.4, and 0.3 cmol₊ kg⁻¹, respectively; and cation exchange capacity (CEC) 21.6 cmol₊ kg⁻¹.

2.2 | Experimental design and treatment characterization

The experiment was arranged in a completely randomized design, considering the plot's high homogeneity, and included eight treatments with three replicates each. Four N rates were applied: 50, 100, 150 and 200 kg ha⁻¹, corresponding to treatments N50, N100, N150 and N200, respectively. Ammonium nitrate fertilizer (27% N, 50% N-NH₄⁺ and 50% N-NO₃⁻) was used as the N source. Furthermore, three additional treatments were included, involving the application of organic amendments: cow manure (CM200), sheep manure (SM200) and sewage sludge (SS200), applied at a rate equivalent to the application of 200 kg ha⁻¹ of N. The amounts of fresh material to be applied were calculated considering the moisture content and N concentration in the dry matter. The eighth treatment corresponded to an unfertilized control (N0).

Cow and sheep manures resulting from mixing animal faeces and urine with food scraps and bedding, typically made from rye straw, were sourced from on-site stables and then composted. The sewage sludge is a material provided

by a local wastewater treatment plant (WWTP). It is derived from a process that involves primary settling and medium-load activation in a liquid line, thickening, hot digestion (35°C) and mechanical dewatering. The average composition of the organic amendments is presented in Table 1.

2.3 | Management of the field experiment

The initial soil tillage was carried out using a mouldboard plough to a depth of 30 cm, followed by a pass with a cultivator at 15 cm. Subsequently, the experimental plots were marked, each with 6 m × 5 m dimensions, and the organic amendments and mineral fertilizer were applied. The organic amendments were applied entirely at sowing, while the mineral fertilizer was applied half at sowing and half as a side-dressing for all four treatments, N50, N100, N150 and N200. After the amendments and mineral fertilizer were manually and evenly applied across the entire surface of the respective plots, they were incorporated into the soil with a final pass of the cultivator. Seeding was done using a single-seed drill, releasing seeds 0.7 m between rows and 0.15 m within rows (95,238 seeds ha⁻¹). Seeding was conducted on May 17, 2022 and May 10, 2023.

During the growing season, the crop was irrigated using a center pivot system, receiving a total of approximately 3000 m³ of water per year, estimated using randomly placed rain gauges in the cultivation field. Weed control was achieved using the herbicide Laudis® (containing 44 and 22 g L⁻¹ of tembotrione and isoxadifene-ethyl, respectively), applied at a concentration of 0.6 L hL⁻¹ at growth stage 14 (4 unfolded leaves) (Meier, 2018). The second half of the mineral fertilizer was applied as a side-dressing on June 20 and June 28 in 2022 and 2023, respectively, at growth stage 16 (6 unfolded leaves) (Meier, 2018). Harvesting was performed at phenological stage 79 (nearly all kernels have reached final size) (Meier, 2018) on September 6, 2022 and August 25, 2023, respectively. The plants were cut with a knife 10 cm above the soil surface and no residues were left on the ground, simulating maize harvesting for silage.

TABLE 1 Selected properties (average \pm standard deviation, $n=3$) of sewage sludge, cow, and sheep manures applied as organic amendments in the experiment.

Properties	Sewage sludge		Cow manure		Sheep manure	
	2022	2023	2022	2023	2022	2023
Moisture (%)	78.9 \pm 4.0	82.0 \pm 7.5	60.6 \pm 1.7	63.2 \pm 7.4	56.7 \pm 7.2	57.3 \pm 3.7
¹ Organic carbon (g kg ⁻¹)	401.4 \pm 2.9	358.1 \pm 10.3	435.2 \pm 12.2	415.3 \pm 15.9	424.7 \pm 6.7	423.7 \pm 7.6
² pH(H ₂ O)	7.7 \pm 0.2	8.0 \pm 0.2	9.2 \pm 0.1	9.1 \pm 0.2	9.2 \pm 0.5	9.3 \pm 0.2
³ Nitrogen (g kg ⁻¹)	48.5 \pm 3.2	49.6 \pm 1.2	22.8 \pm 2.4	24.1 \pm 0.5	31.6 \pm 1.6	29.0 \pm 2.1
⁴ Phosphorus (g kg ⁻¹)	11.2 \pm 2.2	16.8 \pm 1.3	6.3 \pm 1.3	5.7 \pm 0.4	12.1 \pm 2.0	10.2 \pm 0.8
⁴ Boron (mg kg ⁻¹)	12.5 \pm 0.7	20.2 \pm 1.9	27.8 \pm 2.3	25.8 \pm 0.6	43.7 \pm 9.1	42.0 \pm 11.5
⁵ Potassium (g kg ⁻¹)	1.6 \pm 0.3	2.5 \pm 0.6	31.7 \pm 4.1	29.0 \pm 8.0	37.9 \pm 9.6	37.7 \pm 9.9
⁶ Calcium (g kg ⁻¹)	7.2 \pm 0.5	20.2 \pm 3.0	8.8 \pm 0.6	9.3 \pm 1.3	15.1 \pm 0.2	13.6 \pm 1.9
⁶ Magnesium (g kg ⁻¹)	2.5 \pm 0.2	4.5 \pm 1.0	8.1 \pm 0.4	8.8 \pm 0.7	9.1 \pm 1.1	8.7 \pm 0.2
⁶ Iron (g kg ⁻¹)	5.7 \pm 1.1	11.6 \pm 3.1	4.4 \pm 0.5	6.3 \pm 2.2	2.2 \pm 1.0	2.2 \pm 1.0
⁶ Manganese (mg kg ⁻¹)	91.0 \pm 7.1	240.5 \pm 26.4	310.6 \pm 8.0	343.1 \pm 37.9	386.4 \pm 19.9	350.0 \pm 31.5
⁶ Zinc (mg kg ⁻¹)	447.5 \pm 62.5	790.8 \pm 114.9	112.6 \pm 9.2	114.7 \pm 6.2	252.5 \pm 139.6	156.8 \pm 4.3
⁶ Copper (mg kg ⁻¹)	92.2 \pm 5.6	250.6 \pm 21.2	28.0 \pm 3.5	32.9 \pm 3.4	26.8 \pm 1.2	24.7 \pm 1.8

Note: Superscript numbers refer to laboratory methods of determination: ¹Incineration; ²Potentiometry; ³Kjeldahl; ⁴Colorimetry; ⁵Flame emission spectrometry; ⁶Atomic absorption spectrophotometry.

2.4 | Soil and plant sampling

Initial soil samples were collected to obtain a general characterization of the experimental plot. Before the side-dress N application in both years, soil samples were also taken for the pre-side-dress soil nitrate test (Magdoff et al., 1984). Soil was sampled again 1 month after the maize growing season to assess the effect of treatments on general soil properties and inorganic N levels, the latter serving as an indicator of the risk of nitrate leaching before the onset of autumn rains. In the initial sampling, composite samples were collected, with 15 sub-samples per composite sample. In the subsequent samplings, to assess the effect of the treatments, 5 sub-samples were collected per composite sample. Soil sampling was conducted at a depth of 0.0–0.20 m using a hand auger.

Leaf sampling was conducted immediately before side-dressing N. Six leaves per plot were harvested, and the youngest fully expanded leaves were selected. These samples were oven-dried at 70°C and ground for subsequent analysis of N concentration. At maize harvest, 10 plants per plot (5 plants in two adjacent rows) were cut from internal rows without border effects. Total samples were weighed fresh and then a subsample of approximately 1 kg, representative of the main sample, was selected and weighed fresh. After that, this sample was oven-dried at 70°C until reaching a constant weight to determine the dry matter percentage and estimate the total plot yield. The dried samples were also ground for elemental composition analysis. Additionally, a subsample of 15 cm of stem from the base of the plants was collected

to determine nitrate concentration and conduct the stalk nitrate test (Binford et al., 1990). These samples were also oven-dried at 70°C.

2.5 | Laboratory analyses

Soil samples collected at the beginning and end of the study were analysed for organic C by wet digestion (Walkley-Black method), pH (H₂O and KCl) by potentiometry (soil: solution ratio of 1:2.5), exchangeable bases (ammonium acetate, pH 7.0, determined by atomic absorption spectrometry), exchangeable acidity (extracted using KCl and quantified through titration with sodium hydroxide) and extractable B (hot water extraction, determined by the azomethine-H method) (Van Reeuwijk, 2002). Extractable P and K were determined by the Egner-Riehm method (Egnér et al., 1960). In the initial soil samples, soil separates were also determined for texture classification using the Robinson pipette method (Van Reeuwijk, 2002). Soil samples designated for the pre-side-dress soil nitrate test and those collected at the end of the study were analysed for inorganic N. Soil extracts were prepared by mixing 20 g of soil with 40 mL of 2 M KCl solution. The suspension was shaken for 1 hour and filtered through Whatman No. 42 filter paper. Nitrate and ammonium concentrations in the extracts were analysed using a UV-Vis spectrophotometer (Baird et al., 2017).

Leaf samples were analysed for N concentration, while samples of whole plants were analysed for 10

TABLE 2 Soil inorganic nitrogen (nitrate-N, NO_3^- -N; ammonium-N, NH_4^+ -N; and inorganic-N, inorg-N) at pre-side-dress soil N application for treatments with 0 (N0), 50 (N50), 100 (N100), 150 (N150), and 200 (N200) kg ha^{-1} of N applied as mineral fertilizer, and 200 kg ha^{-1} of N applied as sewage sludge (SS200), cow manure (CM200), or sheep manure (SM200).

	2022			2023		
	NO_3^- -N	NH_4^+ -N, mg kg^{-1}	Inorg-N	NO_3^- -N	NH_4^+ -N, mg kg^{-1}	Inorg-N
N0	11.3 bc	3.2 c	14.5 c	8.5 d	3.7 bc	12.2 c
N50	12.7 bc	3.5 c	16.2 bc	9.7 d	3.6 c	13.3 bc
N100	14.3 bc	4.4 bc	18.7 bc	10.4 cd	3.5 c	13.9 bc
N150	15.8 ab	4.5 bc	20.2 abc	16.1 abc	5.3 bc	21.4 a
N200	19.8 a	6.3 ab	26.1 a	19.9 a	4.3 bc	24.2 a
SS200	13.7 bc	7.6 a	21.2 ab	17.1 ab	6.1 ab	23.2 a
CM200	11.0 bc	4.4 bc	15.4 bc	11.6 bcd	8.2 a	19.9 ab
SM200	10.0 c	4.7 c	14.7 c	11.6 bcd	7.7 a	19.3 ab
Prob.	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001
St. error	0.99	0.46	1.26	1.27	0.50	1.39

Note: The same letter in columns indicates statistically not different means by the Tukey HSD test ($\alpha = .05$).

essential nutrients. Nitrogen was determined by the Kjeldahl method, P and B by colorimetry, K by flame emission spectrometry, and Ca, Mg, copper (Cu), iron (Fe), zinc (Zn) and manganese (Mn) by atomic absorption spectrometry (Temminghoff & Houba, 2004). These determinations were performed after nitric digestion of the tissues using microwaves. Nitrate concentration in stalk samples was determined using 1 g of dry tissue, shaken in 50 mL of water, filtered through Whatman No. 42 filter paper and the nitrate concentration was analysed using a UV-Vis spectrophotometer.

2.6 | Data analysis

The data analysis was conducted using SPSS Statistics software (v. 25, IBM SPSS, Armonk, NY). Normality assumptions were checked using the Shapiro-Wilk test and homogeneity of variances was assessed using the Levene test. Subsequently, a one-way ANOVA was performed to identify treatment differences. When significant differences were found, mean separation was performed using the post hoc Tukey HSD test ($\alpha = .05$).

Apparent N recovery (ANR) was calculated according to the following equation (Dimande et al., 2023):

$$\text{ANR (\%)} = \frac{(\text{N recovered in fertilized or amended plots} - \text{N recovery in unfertilized plots})}{\text{N applied as fertilizer or amendment}} \times 100.$$

3 | RESULTS

Indices of soil available N and plant N nutritional status at the time of pre-side-dress N application.

The levels of inorganic N in the soil just before side-dressing fertilization varied significantly across treatments in both 2022 and 2023 (Table 2). Nitrate N (NO_3^- -N) and total inorganic N (Inorg-N) consistently increased with the N rate applied in the mineral fertilization treatments. Soil NO_3^- -N levels ranged from 11.3 to 19.8 mg kg^{-1} in 2022 and 8.5 to 19.9 mg kg^{-1} in 2023 for the N0 and N200 treatments, respectively. Among the treatments involving organic amendments, when considering the overall results from both trial years, the sewage sludge (SS200) treatment tended to exhibit higher values than the cow manure (CM200) and sheep manure (SM200) treatments. In 2022, total inorganic N in the soil was significantly higher in this treatment than in the SM200 treatment. Inorganic N levels in the soil under the SS200 treatment were comparable to those observed in the N200 treatment. However, it should be noted that at this stage, the N200 treatment had only received 100 kg ha^{-1} of N as pre-plant fertilization.

Leaf N concentration before pre-side-dress soil N application varied significantly between treatments in both growing seasons (Figure 2). Among the mineral fertilization treatments, mean values increased progressively from N0 to N200, with leaf N concentrations of 24.5 and 32.7 g kg^{-1} in 2022 and 27.5 and 38.0 g kg^{-1} in 2023 for the

N0 and N200 treatments, respectively. Organic amendments did not show significant differences among treatments in either year of the trial. When compared with mineral fertilizers, the mean values were lower than those

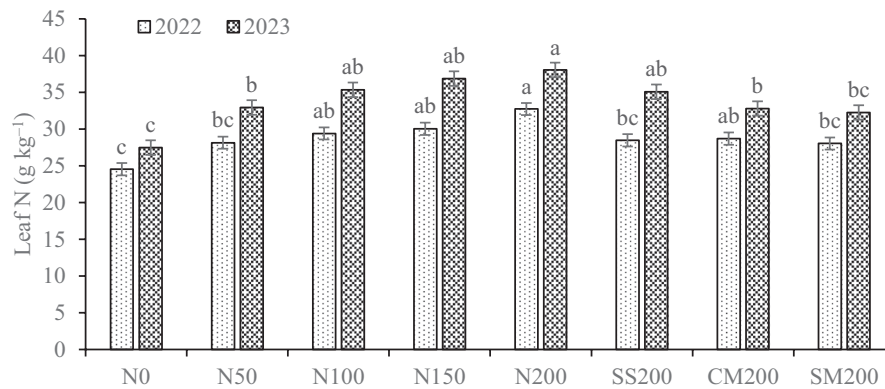


FIGURE 2 Leaf nitrogen (N) at pre-side-dress soil N application for treatments with 0 (N0), 50 (N50), 100 (N100), 150 (N150) and 200 (N200) kg ha⁻¹ of N applied as mineral fertilizer, and 200 kg ha⁻¹ of N applied as sewage sludge (SS200), cow manure (CM200) or sheep manure (SM200). Within each year, means followed by the same letter are not significantly different according to the Tukey HSD test ($\alpha = .05$). Error bars represent standard errors.

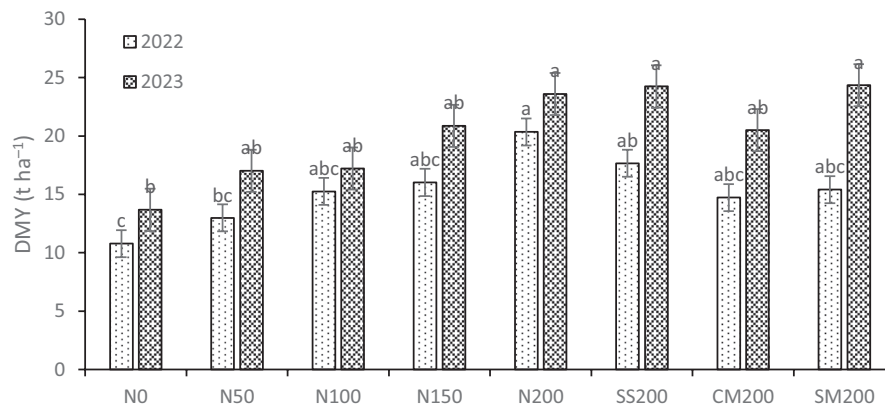


FIGURE 3 Dry matter yield (DMY) for treatments with 0 (N0) 50 (N50), 100 (N100), 150 (N150) and 200 (N200) kg ha⁻¹ of N applied as mineral fertilizer and 200 kg ha⁻¹ of N applied as sewage sludge (SS200), cow manure (CM200) or sheep manure (SM200). Within each year, means followed by the same letter are not significantly different according to the Tukey HSD test ($\alpha = .05$). Error bars represent standard errors.

observed in the N200 treatment but without significant differences. It is important to note that the mineral treatments only received half of the N rate at this stage, corresponding to the basal application. In contrast, the organic fertilizers were fully applied to maize at sowing. Leaf N concentrations in some organic amendment treatments were not significantly different from the control, specifically in SS200 and SM200 in 2022 and SM200 in 2023, although their mean values were numerically higher.

3.1 | Maize DMY and N use efficiency

Maize DMY increased significantly with the mineral N rate (Figure 3). In 2022, mean values ranged from 10.8 to 20.3 t ha⁻¹ for the N0 and N200 treatments, respectively, and from 13.7 to 23.6 t ha⁻¹ in 2023. Dry matter yield did

not vary significantly among organic amendments in 2022 or 2023, though mean values tended to be higher in the SS200 treatment. In 2022, the results for the organic amendments did not differ significantly from the N200 treatment, although mean values were generally lower. Consequently, DMY in the organic amendment treatments also did not differ significantly from the control, except in the SS200 treatment. Nevertheless, the mean values for the organic treatments were higher than those of the control in 2022. In 2023, the results for the organic amendments were generally higher than in 2022 and, in some cases, surpassed those of the higher mineral N rate treatments. Specifically, in 2023, the mean values for the SS200 (24.3 t ha⁻¹) and CM200 (24.4 t ha⁻¹) treatments exceeded those observed in the N200 treatment (23.6 t ha⁻¹).

Nitrogen recovery in the total aboveground biomass of maize increased significantly among the mineral fertilizer

TABLE 3 Nitrogen (N) recovery, apparent N recovery (ANR), stalk nitrate (St nitrate), soil inorganic nitrogen (nitrate-N, NO_3^- -N; ammonium-N, NH_4^+ -N; and inorganic-N, inorg-N) at harvest for treatments with of 0 (N0) 50 (N50), 100 (N100), 150 (N150) and 200 (N200) kg ha^{-1} of N applied as mineral fertilizer and 200 kg ha^{-1} of N applied as sewage sludge (SS200), cow manure (CM200), or sheep manure (SM200).

	2022	2022	2023	2023	2023	2023	2023	2023
	N recovery	ANR	N recovery	ANR	St nitrate	NO_3^- -N	NH_4^+ -N	Inorg-N
	kg ha^{-1}	%	kg ha^{-1}	%	mg kg^{-1}	mg kg^{-1}	mg kg^{-1}	mg kg^{-1}
N0	119.0 c	–	122.4 c	–	263.0 e	11.0 c	7.1 a	18.2 c
N50	159.9 bc	81.6	168.8 bc	92.8	571.3 de	13.1 bc	7.2 a	20.3 bc
N100	192.4 b	73.3	191.7 bc	69.3	1912.9 cd	18.9 bc	7.9 a	26.8 bc
N150	208.7 ab	59.8	264.0 ab	94.4	3146.4 bc	40.5 a	7.8 a	48.3 a
N200	267.0 a	74.0	305.6 a	91.6	4945.8 a	39.9 a	7.8 a	47.7 a
SS200	221.0 ab	51.0	298.5 a	88.1	3703.8 ab	21.9 b	7.9 a	29.8 b
CM200	163.0 bc	22.0	208.3 abc	42.9	2584.5 bc	19.6 bc	6.3 a	26.0 bc
SM200	155.6 bc	18.3	262.1 ab	69.8	2063.5 c	19.2 bc	7.0 a	26.2 bc
Prob.	<.0001		.0001		<.0001	<.0001	.1442	<.0001
St. error	13.97		21.12		279.63	1.00	0.42	2.13

Note: In columns, means followed by the same letter are not statistically different by the Tukey HSD test ($\alpha = .05$); Apparent N recovery (%) = (N recovered in fertilized or amended plots – N recovery in unfertilized plots/N applied as fertilizer or amendment $\times 100$).

treatments, with values ranging from 119.0 to 267.0 kg ha^{-1} in 2022 and from 122.4 to 305.6 kg ha^{-1} in the N0 and N200 treatments, respectively (Table 3). Across both growing seasons, the ANR varied between 59.8% (N150, 2022) and 94.4% (N150, 2023). Nitrogen recovery did not vary significantly among organic amendments, although the SS200 treatment consistently exhibited higher mean values. The N recovery in the SS200 treatment was not significantly different from that in the N200 treatment in either year of the trial. In contrast, the CM200 and SM200 treatments had significantly lower N recovery values than the N200 treatment in 2022. In general, ANR values with organic amendments were lower than those with mineral treatments, particularly in 2022 when the mean ANR for the CM200 treatment was 22.0% and for the SM200 treatment 18.3%.

Nitrate concentrations in maize stalks varied significantly among mineral fertilization treatments, with mean values in 2023 ranging from 263.0 to 4945.8 mg kg^{-1} in the N0 and N200 treatments, respectively (Table 3). Among the organic treatments, the SS200 (3703.8 mg kg^{-1}) had a significantly higher stalk nitrate concentration than the SM200 treatment (2063.5 mg kg^{-1}). When comparing organic amendments with mineral fertilization treatments, the nitrate concentration in the SS200 treatment fell between the values observed in the N150 and N200 treatments, while the values for the CM200 and SM200 treatments were between those of the N100 and N150 treatments.

Soil nitrate levels at the end of the 2023 growing season varied significantly among mineral fertilizer treatments, showing an increasing trend from N0 to

the treatments with higher N rates (N150 and N200) (Table 3). The values for organic amendments were significantly lower than those observed in the higher N rate treatments (N150 and N200) and were comparable to the levels found in the N100 treatment. Ammoniacal N in the soil did not vary significantly among treatments, so total inorganic N followed the same trend as nitrate levels.

3.2 | Macronutrients other than N and micronutrients in plant tissues

Phosphorus concentrations in whole maize plants ranged from 1.48 to 1.71 g kg^{-1} in 2022, with no significant differences between treatments (Table 4). However, in 2023, significant differences were observed, with the lowest values in treatments that received either no fertilizer or low rates of mineral fertilizer and the highest concentration in the SS200 treatment. Potassium concentration in plant tissues varied significantly among treatments in 2022 but not in 2023. In 2022, the lowest values were recorded in the SS200 treatment, while the highest were observed in the SM200 and CM200 treatments. Although no significant differences were found in tissue K concentration in 2023, the trend observed in 2022 persisted. Calcium and Mg concentrations in plant tissues did not vary significantly between treatments in either growing season.

Micronutrient concentrations in plant tissues varied significantly among treatments, except for Zn in 2022 and

	Phosphorus		Potassium		Calcium		Magnesium	
	2022	2023	2022	2023	2022	2023	2022	2023
	g kg^{-1}							
N0	1.66 a	1.30 b	5.87 ab	5.75 a	1.98 a	1.28 a	2.79 a	2.11 a
N50	1.48 a	1.21 b	6.16 ab	5.89 a	1.81 a	1.48 a	2.57 a	2.38 a
N100	1.58 a	1.30 b	5.26 b	6.07 a	1.69 a	1.41 a	2.77 a	1.98 a
N150	1.70 a	1.54 ab	5.66 ab	6.36 a	1.78 a	1.42 a	2.85 a	1.89 a
N200	1.62 a	1.39 ab	5.75 ab	6.26 a	1.51 a	1.19 a	2.70 a	1.72 a
SS200	1.71 a	1.71 a	4.68 b	5.62 a	1.91 a	1.50 a	3.46 a	2.42 a
CM200	1.69 a	1.45 ab	6.38 ab	6.58 a	1.67 a	1.24 a	3.53 a	2.36 a
SM200	1.57 a	1.45 ab	7.53 a	6.94 a	2.01 a	1.31 a	3.75 a	2.16 a
Prob.	.8410	.0091	.0170	.7756	.2590	.3445	.1810	.2769
St. error	0.12	0.08	0.45	0.59	0.14	0.10	0.35	0.21

Note: In columns, means followed by the same letter are not statistically different by the Tukey HSD test ($\alpha = .05$).

Cu in 2023 (Table 5). However, few results were consistent across the two growing seasons, N rates, or between mineral and organic fertilizers. The most consistent finding was a tendency for lower Fe concentrations in tissues associated with applying organic amendments.

3.3 | Soil properties

Soil pH did not vary significantly among treatments (Table 6), with mean values ranging from 6.0 to 6.3. Organic C content in the soil was significantly higher in treatments with organic amendments ($26.0\text{--}28.3 \text{ g kg}^{-1}$) compared with the control and mineral N treatments ($20.2\text{--}21.9 \text{ g kg}^{-1}$). Soil P levels varied significantly among treatments, with the SS200 treatment having the highest mean value, while the control and mineral fertilizer treatments had lower average levels. The SM200 and CM200 treatments showed significantly higher soil K values than the others. Soil CEC was significantly higher in the treatments with organic amendments due to higher Ca and Mg levels across all organic treatments and higher K levels in the SM200 and CM200 treatments.

4 | DISCUSSION

4.1 | Soil available N and plant N nutritional status at side-dressing

Inorganic N levels in the soil before side-dress fertilization significantly increased with higher basal N application rates. Correspondingly, leaf N concentration also consistently rose by the amount of N applied. These are variables commonly

used in monitoring the N available to plants during a crucial phase of the growing season. The soil's inorganic N level before side-dressing, initially assessed using the pre-side-dress soil nitrate test (Magdoff et al., 1984, 1990), along with leaf N concentration are important indicators of soil N availability. They are widely used to inform the side-dress N application rates, given the challenges associated with accurately determining the N requirements of crops based on pre-plant soil tests (Meisinger et al., 2008; Rütting et al., 2018; Weil & Brady, 2017). Since these tests are conducted during the growing season, they provide a more realistic estimate of the soil's available N and the need for supplementary N fertilization (Rodrigues, 2004; Rodrigues et al., 2021; Weil & Brady, 2017).

The results demonstrated that basal mineral N application significantly improved the nutritional status of plants. This is primarily because N was applied as ammonium nitrate ($50\% \text{ NH}_4^+$, $50\% \text{ NO}_3^-$), containing highly soluble forms of N that are readily available for root uptake (Bronson, 2008; Havlin et al., 2017). However, the differences between treatments also reveal the inability of young plants to fully uptake the N applied, leaving a portion of it in the soil. At this time, most of the N was in nitrate form. Even though half of the N was applied as NH_4^+ , it likely underwent nitrification, a relatively rapid process when soil temperature and moisture are adequate (Norton, 2008; Weil & Brady, 2017). This transformation is driven by microbial activity, particularly the *Nitrosomonas* genus, which converts NH_4^+ to NO_2^- , and *Nitrobacter* converts NO_2^- to NO_3^- (Havlin et al., 2017; Weil & Brady, 2017).

The levels of inorganic N in the soil and the N concentration in leaves from treatments with organic amendments did not differ as distinctly from the control as the treatments with higher rates of mineral N, even though

TABLE 4 Tissue macronutrient concentrations for treatments with 0 (N0) 50 (N50), 100 (N100), 150 (N150), and 200 (N200) kg ha^{-1} of N applied as mineral fertilizer and 200 kg ha^{-1} of N applied as sewage sludge (SS200), cow manure (CM200), or sheep manure (SM200).

TABLE 5 Tissue macronutrient concentrations for treatments with 0 (N0) 50 (N50), 100 (N100), 150 (N150), and 200 (N200) kg ha⁻¹ of N applied as mineral fertilizer and 200 kg ha⁻¹ of N applied as sewage sludge (SS200), cow manure (CM200) or sheep manure (SM200).

	Iron		Manganese		Zinc		Copper	
	2022	2023	2022	2023	2022	2023	2022	2023
	mg kg ⁻¹							
N0	154.7 ab	140.3 a	32.5 a	19.8 ab	22.9 a	27.5 a	5.8 ab	7.4 a
N50	189.4 a	116.3 ab	26.6 ab	18.7 b	20.4 a	23.5 ab	6.1 ab	8.7 a
N100	172.3 a	108.2 b	32.1 a	20.5 ab	22.3 a	26.6 a	6.1 ab	8.0 a
N150	124.4 abc	99.6 bc	24.5 ab	27.5 ab	28.1 a	22.4 ab	6.5 a	9.0 a
N200	111.0 abc	104.7 b	20.3 b	28.2 a	23.4 a	25.0 ab	5.2 ab	7.6 a
SS200	87.5 bc	68.9 d	26.7 ab	21.1 ab	27.4 a	20.0 ab	6.5 a	8.0 a
CM200	61.8 c	77.3 cd	24.6 ab	24.6 ab	20.8 a	18.5 b	4.5 b	6.9 a
SM200	82.1 bc	72.4 d	26.2 ab	22.3 ab	23.0 a	17.5 b	6.1 ab	7.4 a
Prob.	.0010	<.0001	.0340	.0139	.5140	.0028	.0200	.0539
St. error	17.32	5.51	2.34	1.84	2.91	1.61	0.36	0.44

Note: In columns, means followed by the same letter are not statistically different by the Tukey HSD test ($\alpha = .05$).

TABLE 6 Soil pH (H₂O), organic carbon (OC), extractable phosphorus (as P₂O₅), and potassium (as K₂O), exchangeable calcium (Ca²⁺), magnesium (Mg²⁺), potassium (K⁺), and sodium (Na⁺) and cation exchange capacity (CEC) for treatments with 0 (N0) 50 (N50), 100 (N100), 150 (N150) and 200 (N200) kg ha⁻¹ of N applied as mineral fertilizer and 200 kg ha⁻¹ of N applied as sewage sludge (SS200), cow manure (CM200) or sheep manure (SM200).

	pH (H ₂ O)	OC g kg ⁻¹	Extractable		Exchangeable				
			P (P ₂ O ₅)	K (K ₂ O)	Ca ⁺⁺	Mg ⁺⁺	K ⁺	Na ⁺	CEC
			mg kg ⁻¹		cmol _c kg ⁻¹				
N0	6.3 a	21.4 b	118.6 c	80.5 b	11.9 c	6.6 cd	0.2 b	0.2 a	19.0 bc
N50	6.2 a	20.9 b	104.6 c	78.0 b	11.5 c	6.2 d	0.2 b	0.3 a	18.3 c
N100	6.2 a	20.2 b	99.6 c	92.0 b	11.8 c	6.3 d	0.2 b	0.4 a	18.7 bc
N150	6.0 a	21.9 b	126.2 bc	99.7 b	12.4 c	6.2 d	0.3 b	0.2 a	19.2 bc
N200	6.0 a	21.5 b	117.6 c	103.1 b	13.3 bc	7.1 bcd	0.3 b	0.2 a	20.9 b
SS200	6.2 a	26.4 a	167.7 a	96.0 b	16.2 a	7.6 abc	0.3 b	0.2 a	24.4 a
CM200	6.3 a	26.0 a	135.7 abc	148.5 a	15.0 ab	8.2 ab	0.5 a	0.2 a	23.9 a
SM200	6.2 a	28.3 a	158.4 ab	153.3 a	15.9 a	8.4 a	0.6 a	0.2 a	25.1 a
Prob.	.1891	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	.0508	<.0001
St. error	0.08	0.80	7.09	5.24	0.34	0.23	0.04	0.04	0.46

Note: In columns, means followed by the same letter are not statistically different by the Tukey HSD test ($\alpha = .05$).

the latter had only received half of the total dose (i.e., the basal application). This occurs because organic amendments release nutrients gradually over time as mineralization takes place (Myrold & Bottomley, 2008; Weil & Brady, 2017). Given the short time frame between the application of organic amendments and this evaluation, it is expected that the plants would show a lower response and that N availability in the soil would be less compared to the application of ammonium nitrate, which is immediately available to plants. Even throughout an entire

growing season, organic amendments typically release less than half of their nutrients (Arrobas et al., 2022; Mallory et al., 2010; Rodrigues et al., 2006, 2018).

4.2 | Dry matter yield, N recovery and N use efficiency

Dry matter yield increased significantly and progressively with the application of mineral N both in the first and second

years of the trial. Similarly, the amount of N recovered in the aboveground biomass of maize also rose significantly with higher N rates. Nitrate levels in the maize stalks at harvest sharply increased from the control to the highest N rate. Additionally, nitrate concentrations in the soil at harvest were significantly higher with increasing N application rates. Mineral N fertilization often boosts crop production, particularly for high nutrient-removal crops like maize (Córcoles et al., 2020; Davies et al., 2020; Yan et al., 2021). When high N rates are applied, it accumulates in plant tissues as nitrates. Ammonia uptake requires rapid assimilation, as high NH_3 levels are toxic to plant tissues (Hawkesford et al., 2023). Maize stalks can store significant amounts of unmetabolized N in the form of nitrates concentrated in the vascular bundles (Isla et al., 2015; Rodrigues et al., 2006, 2018; Yang et al., 2017). This phenomenon prompted the development of the stalk nitrate test (Binford et al., 1990), which, although a post-mortem test, provides valuable information regarding the N fertilization program of the current year. This data can be used to adjust fertilization strategies for the following year (Isla et al., 2015; Rodrigues et al., 2006, 2021; Yang et al., 2017). Maize is typically considered to have received adequate N rates when stalk nitrate levels are within the range of 700–2000 mg kg^{-1} . Values above this range indicate excessive N fertilization (Blackmer & Mallarino, 1996). In this study, treatments with the highest N rates and the SS200 treatment exhibited nitrate levels well above the upper limit of this range, which increases the risk of N losses through nitrate leaching and denitrification.

This inorganic N, often referred to as residual N, is particularly vulnerable to leaching and denitrification in temperate climates where the maize cycle is followed by a cold, rainy season (Randal et al., 2008; Raun & Schepers, 2008). Various complementary strategies, beyond simply reducing N rates, have been developed to minimize N losses from the soil. Among the most ecologically significant strategies is growing winter cover crops or catch crops, which can develop during the winter and help retain N within the system (Notaris et al., 2018; Rodrigues et al., 2021; Valkama et al., 2015).

Dry matter yield in treatments with organic amendments was generally lower in the first year than in the treatments with an equivalent rate of inorganic N. However, in the second year, production levels were very similar, indicating a positive cumulative effect that was less evident in the inorganic fertilizer treatments, although overall production in the second year was higher across the board. The amount of N recovered in the aboveground biomass and, consequently, the ANR was substantially higher in the second year for organic treatments. However, inorganic N levels in the soil at the end of the second growing season tended to be lower in these treatments than those receiving higher mineral fertilizer rates.

In this study, organic amendments provided less N to plants during the growing season than mineral fertilizer treatments with equivalent N rates. This can be attributed to the fact that nutrients from organic amendments only become available to plants after the mineralization of organic matter, a process that occurs gradually over time (Beegle et al., 2008; Myrold & Bottomley, 2008; Weil & Brady, 2017). The decomposition of organic substrates follows a process known as the decay series, in which a relatively large portion mineralizes in the first year, with progressively smaller amounts released in subsequent years (Havlin et al., 2017; Myrold & Bottomley, 2008). However, with recurring applications, the second year sees the mineralization of both the second-year fraction from the first application and the first-year fraction from the second application. This creates a cumulative effect in releasing nutrients from previous years' organic amendments (Daudén et al., 2004; Mallory et al., 2010; Rodrigues et al., 2018). In this study, this cumulative effect likely explains the improved performance of organic amendments in enhancing maize DMY during the second year.

The results for sewage sludge exhibit notable differences from the other organic amendments that are worth highlighting. Although not statistically significant, maize DMY tended to be higher when amended with sewage sludge. Similarly, N recovery and ANR displayed consistently higher average values in both cropping cycles for the SS200 treatment, with particularly notable increases in nitrate content in maize stalks. When considering inorganic N levels in the soil and leaf N concentration at pre-side-dressing, the average values for SS200 also tended to surpass those of the other two organic amendments. Apart from environmental factors like temperature, the key factor determining an organic substrate's decomposition rate is its C/N ratio (Beegle et al., 2008; Myrold & Bottomley, 2008; Weil & Brady, 2017). Among the three organic amendments used, sewage sludge had the lowest C/N ratio (7.2–8.3) compared to cow manure (17.2–19.1) and sheep manure (13.4–14.6). Furthermore, while animal manures often contain significant amounts of cereal straw used as bedding material, composed of lignin, cellulose and hemicellulose, which are low-energy compounds for microorganisms and difficult to decompose (Havlin et al., 2017; Myrold & Bottomley, 2008), sewage sludge consists of more easily degradable materials, which accelerates the rate at which nutrients are released (Arrobas et al., 2024; Rodrigues et al., 2024).

4.3 | Effect of treatments on the bioavailability of nutrients other than N

The results indicated a tendency for elevated P levels in the SS200 treatment, along with low K levels and

generally low Fe concentrations across treatments involving organic amendments. The consistently high P levels observed in the SS200 treatment may be attributed to the initially elevated P content in this organic amendment and its rapid mineralization, a pattern documented in previous studies (Arrobas et al., 2024; Rodrigues et al., 2024). Sewage sludge typically contains low levels of K, as K does not form part of organic structures in plants (Hawkesford et al., 2023) and being highly water-soluble, it is not retained in the solid fraction of sewage sludge, instead being lost in the wastewater.

The low Fe levels in treatments with organic amendments may be due to improved soil aeration, a common outcome of applying organic amendments and increasing organic matter content in the soil (Beegle et al., 2008; Myrold & Bottomley, 2008; Weil & Brady, 2017). Well-aerated conditions typically reduce the solubility and plant availability of Fe, as anoxic conditions increase Fe solubility and its subsequent uptake by plants (Afonso et al., 2020; Alhdad et al., 2015).

4.4 | Soil properties after the end of the second growing season

The fertilization treatments consistently influenced soil organic C levels, with significantly higher values observed in treatments with organic amendments than in the control and mineral fertilizer treatments. Notably, soil P levels tended to be higher, while K levels were significantly lower in the SS200 treatment compared to the other organic amendments. Organic amendments also stood out from mineral fertilizer treatments by increasing CEC due to elevated exchangeable Ca and Mg levels across all organic amendment treatments and increased K levels in the CM200 and SM200 treatments. One commonly advocated reason for the preferential use of organic amendments over mineral fertilizers is their tendency to enhance soil organic matter content, a claim that has been widely substantiated by experimental research (Arrobas et al., 2022; Dimande et al., 2023; Menino et al., 2021; Su et al., 2021). Organic matter, in turn, improves various physical, chemical and biological soil properties that promote plant growth (Weil & Brady, 2017). The variation observed in P and K levels in both plant tissues and soil appears to be a direct result of the initial composition of each organic amendment. Organic amendments also contributed to the increase in CEC by supplying Ca and Mg, the most quantitatively significant exchangeable bases. Furthermore, humic substances, such as humic and fulvic acids, enhance the negative charge of soils, playing a critical role in the adsorption of these cations (Weil & Brady, 2017; Yang et al., 2024).

5 | CONCLUSIONS

Inorganic N fertilization significantly increased soil inorganic N levels and leaf N concentrations during side-dressing compared with organic amendments. This enhancement is attributed to the immediate availability of ammonium and ions, in contrast to the slow-release nature of organic amendments. In the first year, the higher N availability from mineral fertilizers resulted in increased DMY of maize, greater N recovery and improved N use efficiency. The lower N use efficiency observed with organic amendments is unlikely to have caused significant environmental contamination, as a substantial portion of the N likely remained in organic form without undergoing mineralization.

In the second year, an accumulated effect of N release from the organic amendments was noted, leading to maize DMY comparable to those with the highest rates of applied mineral N. However, this was associated with lower stalk nitrate concentrations and reduced inorganic N levels in the soil. These findings suggest a reduced risk of environmental contamination associated with using organic amendments compared to mineral N fertilizers. Nevertheless, if the application of organic amendments continues over time, the cumulative N release from successive applications may negate the previously observed benefits. This indicates that the quantities of organic amendments applied to crops must also be carefully monitored to ensure that environmental contamination remains low.

Sewage sludge exhibited behaviour intermediate between mineral fertilizers and conventional farmyard manures, mineralizing rapidly and having a more immediate effect on vegetation. This endows it with high agronomic value, reflected in a positive effect on plant nutritional status and DMY. This information is valuable for promoting the rational use of this important fertilizing resource in agriculture.

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CONFLICT OF INTEREST STATEMENT

The authors declare no conflicts of interest.

DATA AVAILABILITY STATEMENT

The original contributions presented in the study are included in the article, further inquiries can be directed to the corresponding author.

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