

Effectiveness of zeolite, biochar, and dicyandiamide in reducing nitrogen losses (N₂O emissions and NH₃ volatilizations) from farming dairy effluent under drought

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ABSTRACT: The combination of farming dairy effluent (FDE) with mitigating agents' nitrogen (N) losses to the environment, such as zeolites, dicyandiamide (DCD), and biochar, may reduce losses and improve N availability to plants. A field experiment was conducted in an Argiudol of the Pampa region, Argentina, to evaluate N use efficiency and associated losses following FDE application. On a natural grassland with a predominance of ryegrass (*Lolium multiflorum*) and the presence in smaller quantities of fescue (*Festuca* sp.), alfalfa (*Medicago sativa*), and white clover (*Trifolium repens*). The treatments included application of FDE, FDE plus zeolite (Zeo), FDE plus dicyandiamide (DCD), FDE plus biochar (BC), and a control untreated soil (C), each with three replicates. A uniform application of 11 L m⁻² of effluent (eq to 250 kg of N ha⁻¹) was performed, and nitrous oxide (N₂O) emissions, ammonia (NH₃) volatilization, soil and atmospheric temperature, and water-filled pore space were monitored up to three months. Overall, N₂O emissions were low across all treatments, reaching a maximum of 18.4 µg m⁻² h⁻¹ of N-N₂O. Zeo and BC significantly reduced N₂O emissions by 63.1 and 63.8 %, respectively, compared with FDE alone. Furthermore, BC reduced NH₃ volatilization by 55.5 % relative to FDE, highlighting its strong potential to mitigate total N losses. Regarding pasture performance, the highest greenness index was observed in the BC treatment, followed by intermediate values in FDE, DCD, and Zeo, and the lowest in the control. Under our study conditions, with particularly low rainfall and high temperatures, the mitigating effects of DCD and Zeo appeared to be limited. In contrast, the combination of FDE and BC emerged as the most effective strategy to reduce N losses and enhance pasture quality. Furthermore, environmental conditions play a key role in determining the efficacy of mitigation strategies to curb N losses.

Keywords: nutrient management, mitigating agents, organic fertilizer.

INTRODUCTION

Nitrogen (N) fertilization is an effective way to enhance productivity and improve pasture quality, which in turn can lead to better milk production (Beukes et al., 2020). However, the increase in dairy waste poses an environmental challenge (Milani et al., 2011). One potential solution is to utilize farm dairy effluent (FDE) as an organic fertilizer (Iocoli et al., 2021; Cosentino et al., 2024). The efficiency of FDE can be further improved by incorporating nitrification inhibitors (NI) or materials like zeolites or biochar, which help to increase nitrogen use efficiency (NUE) by releasing N gradually (He et al., 2025). Ammonia volatilization is a main pathway of nitrogen loss, and in the case of organic fertilizers, it can account for up to 64 % of the total nitrogen applied, depending on the type of material, application method, and environmental conditions (Wester-Larsen et al., 2022).

On the other hand, nitrous oxide (N_2O) has a global warming potential 273 times higher than carbon dioxide (CO_2) and is one of the leading greenhouse gases (GHG) emitted by the agricultural sector (Forster et al., 2021). In this sense, delivering nutrients from mineral or organic fertilizers out of sync with crop uptake causes inefficiencies with the risk of environmental contamination due to NH_3 volatilization, N_2O emissions, or nitrate leaching (NO_3^-) (Chin et al., 2018). Consequently, reducing N losses in agricultural systems is a major challenge for modern agriculture. The recovery of FDE and its use as a source of N for crops and pastures, maximizing NUE, is undoubtedly a sustainable alternative, providing a solution to waste disposal and contributing to reducing the use of synthetic fertilizers (Cosentino et al., 2024).

Nitrification inhibitors (NIs) are chemical compounds that suppress the first step of nitrification by inhibiting a broad range of ammonia-oxidizing organisms, including *Nitrosomonas* spp. (McCarty, 1999; Lei et al., 2022). Their use promotes nitrogen retention in the form of ammonium (NH_4^+), which is less mobile in the soil solution, while reducing nitrate (NO_3^-) concentrations and thus the substrate available for denitrification and N_2O production (Di et al., 2014; Gonzatto et al., 2016). Among them, dicyandiamide (DCD) stands out as a widely used NI due to its low cost, water solubility, low volatility, and effectiveness when applied with nitrogen fertilizers (Di et al., 2011). Several studies have demonstrated that NIs, including DCD, can significantly reduce N_2O emissions from soils (Di et al., 2012; Gonzatto et al., 2016). However, limited information is available regarding their effectiveness when applied with organic waste, particularly farm dairy effluent (FDE). Evaluating the impact of DCD on N_2O emissions and NH_3 volatilization from FDE-treated soils is essential for identifying management strategies that enhance productivity while reducing environmental losses.

Application of FDE with zeolite represents an interesting strategy, as zeolites are low-cost minerals with a microporous structure and high cation exchange capacity (Park et al., 2024). These properties allow zeolite to retain nutrients and reduce the mobility of potentially toxic elements (PTEs) by adsorbing them onto its surface (Wang and Peng, 2010; Mohsin et al., 2023). Zeolites can also modulate nutrient release and regulate soil moisture content (Eroglu et al., 2017), which may help reduce N losses, particularly N_2O emissions, when co-applied with organic amendments such as FDE.

Another product with GHG mitigation potential is biochar (Doyeni et al., 2023), a porous carbonaceous material created through the pyrolysis of organic matter in low-oxygen conditions. This material features a microporous structure, which gives a high cation exchange capacity and a diverse range of physical and chemical properties, influenced by factors such as the type of raw material used, the conditions of pyrolysis, and the nutrient content in the biochar (Niraula et al., 2021). Like zeolite, biochar can retain and diminish the bioavailability of PTE, while also gradually adsorbing and releasing nutrients through the mineralization of FDE (Peng et al., 2020; Zhang et al., 2023). Additionally, biochar has significant potential for sustainable agriculture by helping to reduce GHG emissions (Zhang et al., 2012). Nonetheless, the effectiveness of biochar application in agricultural settings is greatly influenced by factors such as soil type, the biochar-to-soil

ratio, and climatic conditions. Consequently, further research is necessary to optimize biochar use in agriculture.

The evaluation of FDE management strategies amended to the soil is critical to reduce nitrogen losses to the environment, improve pasture performance and quality, enhancing soil and air health. It was hypothesized that (i) the combination of FDE with these amendments reduces nitrogen losses as N_2O and NH_3 compared with FDE alone, and (ii) these combinations improve pasture performance. This study aimed to evaluate different management strategies to mitigate atmospheric nitrogen losses by applying FDE combined with a nitrification inhibitor (DCD), zeolite, and biochar under drought conditions.

MATERIALS AND METHODS

Field experiment

The field experiment was carried out on a natural grassland without animal, with predominance of ryegrass (*Lolium multiflorum*) and presence in smaller quantities of fescue (*Festuca* sp.), alfalfa (*Medicago sativa*), white clover (*Trifolium repens*) located in Castelar, Buenos Aires province, Argentina (34°37'21.13" S - 58° 40'12.44" W), from October 12, 2022, to January 13, 2023. The soil in the area is classified as Vertic Argiudol (Soil Survey Staff, 2022) with a silty loam texture (sand 165 g kg⁻¹, silt 585 g kg⁻¹, and clay 250 g kg⁻¹). Soil had a value of 6.3; electrical conductivity of the saturated paste extract was 0.2 dS m⁻¹; percentage of organic carbon of 1.8; percentage of N of 0.2; and bulk density of 1.20 Mg m⁻³ in the first 0.20 m of soil.

Treatments included application onto soil of farm dairy effluent alone (FDE), FDE plus zeolite (Zeo), FDE plus dicyandiamide (DCD), FDE plus biochar (BC), and the control untreated (C). A completely randomized design was used, with three replicates. The FDE was collected fresh two days before applying it to the soil from a stabilization lagoon of a milk farm in the town of Lobos, Buenos Aires province. The FDE was stored in a cool, shady place, in 100-liter plastic drums. The characterization of the FDE is presented in table 1. Total carbon and nitrogen, and carbon-to-nitrogen ratio (C/N) of each treatment material were determined before field application and are shown in table 2. These values provide additional information on the biochemical characteristics of the amendments, which could influence nitrogen dynamics.

The biochar was produced through a pyrolysis process (100 min at 600 °C) using a species of bamboo cane called Tacuaruzú (*Guadua chacoensis*) as an input. It was ground by hand and sieved through two mesh screens, size 20 and 4 (corresponding to a particle size between 0.84 and 4.75 mm). The zeolites used belong to the clinoptilolite-heulandite series and were obtained in the province of La Rioja, Argentina. They were supplied and characterized by the Argentine Geological and Mining Service (SEGEMAR) and processed by grinding and sieving through a 200 ASTM mesh screen, resulting in particles with a size of 0.074 mm or less.

In all treatments involving effluent, a volume equivalent to 11 L effluent m⁻² (corresponding to 250 kg ha⁻¹ of N) was applied superficially and uniformly across each experimental plot 12 m² (3 × 4 m). The amount of effluent was carefully adjusted according to the area of each measurement device (N_2O chambers and NH_3 towers) and applied manually to ensure accurate dosing in these critical measurement zones.

We applied zeolite at 100 g L⁻¹ FDE (~11 Mg ha⁻¹), biochar at 15 g L⁻¹ of FDE (≈ 1.66 Mg ha⁻¹), and dicyandiamide (DCD) at 10 kg ha⁻¹. Zeolite and biochar were pre-mixed into the effluent 24 h before soil application, while DCD was co-applied directly with the FDE at the time of application. These rates were selected based on prior studies showing agronomic and nitrogen-related benefits at comparable doses: 10–15 Mg ha⁻¹ zeolite improved N retention and use efficiency across crops (GirijaVenii et al., 2021; Hazrati et al., 2022); ≤ 3 Mg ha⁻¹ biochar proved effective as a low dose in temperate field

conditions (Jiang et al., 2023), and 0.5–4.0 % was effective in pot trials (Qiu et al., 2022); and 10 kg ha⁻¹ DCD was supported by field trials in the Pampas under cool, moist conditions (Cosentino et al., 2024), as well as mechanistic evidence indicating this dose can significantly reduce nitrification (Guo et al., 2014). All rates were later refined and validated through unpublished trials under our local environmental conditions.

The grass was cut to a height of 0.05 m above the ground, five days before the experiment began, to homogenize initial conditions, and was only cut to assess pasture yield. During the experimental period, the average soil temperature was 16.3 °C, the air temperature averaged 29.3 °C, and accumulated precipitation totalled 142.2 mm.

Table 1. Characterization of the FDE for the treatments farm dairy effluent (FDE), farm dairy effluent plus zeolite (Zeo), farm dairy effluent plus Dicyandiamide (DCD) and farm dairy effluent plus biochar (BC)

Property	Zeo	FDE	DCD	BC
pH	8.2	8.1	8.1	6.8
Electrical conductivity (dS m ⁻¹)	5.4	5.8	5.9	4.6
Total solids (g L ⁻¹)	161.3	78.8	78.5	108.4
Volatile solids (g L ⁻¹)	34.5	33.8	33.8	38.8
Organic C (g L ⁻¹)	3.5	23.8	23.7	10.6
Total N (g L ⁻¹)	0.28	2.23	2.23	1.69
Phosphorus (g L ⁻¹)	0.15	0.25	0.25	0.22
Potassium (g L ⁻¹)	1.27	1.4	1.4	1.09
Calcium (g L ⁻¹)	1.45	0.72	0.72	0.92
Magnesium (g L ⁻¹)	1	0.43	0.44	0.55
Sulfur (g L ⁻¹)	0.31	0.33	0.33	0.37
Boron (mg L ⁻¹)	2.4	0.7	0.7	1
Ammonium (mg L ⁻¹)	63	441	441	366
Nitrate (mg L ⁻¹)	1	5	5	3
BOD ₅ (mg L ⁻¹)	<5.000	<5.000	<5.000	<5.000
COD (mg L ⁻¹)	34.000	34.000	34.000	34.000
Salmonella	Not detected	Not detected	Not detected	Not detected
<i>E. Coli</i> (NMP 100 mL ⁻¹)	<1.600	<1.600	<1.600	<1.600

BOD₅: 5-day biological oxygen demand; COD: chemical oxygen demand.

Table 2. Total N and C contents, and the C/N ratio of the pasture, for the treatments farm dairy effluent (FDE), farm dairy effluent plus zeolite (Zeo), farm dairy effluent plus Dicyandiamide (DCD), farm dairy effluent plus biochar (BC), and control (C)

Treatment	C	N	C/N ratio
	%	%	
C	53.5	1.4	38.1
FDE	51.0	1.3	40.2
BC	51.4	1.6	35.1
Zeo	51.8	1.4	37.3
DCD	49.5	1.3	40.5

N₂O emission

The static chamber method was used for sampling N₂O during the 92 days after application (DAA). Each static chamber consisted of a metal base covering a surface area of 0.13 m² and a non-vented plastic top with a height of 0.125 m. The metal base was inserted into the soil to a depth of 0.05 m soon after cutting the ryegrass to avoid possible gas leakage or contamination by lateral diffusion (Clough et al., 2020). Sampling was carried out in the morning (between 09:00 am and 12:00 pm), as described by Cosentino et al. (2012). Gas samples were taken from the chamber headspace at 0, 15, and 30-min after closing the chambers. Gases were extracted directly from inside the chamber (volume 16.7 dm³) using a vacuum pump and injected into previously evacuated 10 cm³ vials sealed with rubber stoppers fixed to the vial with an aluminum flange (Alves et al., 2012; Cosentino et al., 2012), we followed this procedure on each sampling date and for each of the 3 chambers within each plot. Within seven days of sampling, N₂O was measured in the laboratory with a GC 6890 Agilent Technologies Network gas chromatograph, fitted with a 63Ni electron capture detector (Agilent Network GC System, A' ECD, Santa Clara, CA, USA) and a 30 m × 530 μm × 25 μm Molsieve HP-Plot column. The oven, injector and detector temperatures were 150, 100 and 300 °C, respectively. The carrier gas was N₂, and the injection volume was 0.5 cm³. The N₂O fluxes (*f*) were calculated using equation 1.

$$f = \frac{\Delta C}{\Delta t} \times \frac{V}{A} \times \frac{m}{V_m} \quad \text{Eq. 1}$$

in which: $\Delta C/\Delta t$ is the change in N₂O concentration in the chamber during the deployment time Δt ; *V* is the volume of the chamber (16.7 dm³); *A* is the soil area (0.13 m²) covered by the chamber; *m* is the molecular mass of N₂O; and *V_m* is molar volume of N₂O. The N₂O emission fluxes were calculated by the linear regression method, taking the accumulation time (*t*₀, *t*₁₅, *t*₃₀ min) as the independent variable and the N₂O concentration in each of those as the dependent variable times (*C*_{*t*0}, *C*_{*t*15}, *C*_{*t*30}).

NH₃ volatilization

The static semi-open-cylinder absorption system proposed by Nômmik (1973) and modified by Videla (1994) was used to measure NH₃ volatilization during the 22 DAA. This system featured a cylindrical collection tower with polyethylene walls, measuring 0.24 m in diameter and 0.80 m in height. Within the tower, two metal rods supported two plates of expanded polyurethane, each 1.5 cm thick, saturated with a 0.25 mol L⁻¹ H₂SO₄ solution. The lower plate, positioned 0.15 m above the soil, captured NH₃ volatilization from the soil, while the upper plate acted as a barrier to prevent contamination from atmospheric NH₃. Additionally, each tower was outfitted with a zinc roof to prevent rainwater from entering. Ammonia determinations over time were carried out until no significant differences (*p*<0.05) were observed between the treatments and the control (Videla, 1994). This gas was sampled daily for the first nine days after applying the treatments, then once a week for two weeks. In each sampling, the polyurethane plate of the lower grid was rinsed with distilled water, and the NH₄⁺ content in the sponge-washing solution was determined by the micro distillation method and subsequent titration (Bremner and Keeney, 1965). From the N-NH₄⁺ value, the concentration of N-NH₃ volatilized over the surface covered by the NH₃ collection chamber was obtained as nitrogen capture (*N_c*), according to equation 2.

$$N_c \text{ captured in the cylinder} = (V_a - V_b) \times [Nac / (V_m \times m \times 500)] \quad \text{Eq. 2}$$

in which: *V_a* is the volume of sulfuric acid used for the sample (cm³); *V_b* is the volume of sulfuric acid used for the blank (cm³); *Nac* is the normality of the sulfuric acid (meq cm⁻³); *V_m* is the volume of the sample (cm³); and *m* is the weight of the meq of N.

Nitrogen lost due to volatilization of N-NH_3^+ per hectare (N_v) was calculated according to the equation 3.

$$N_v = (N_c \times 10000) / CS \quad \text{Eq. 3}$$

in which: N_c is the N from NH_3 captured by the cylinder; 10000 is the area of one hectare in (m^2); and CS is the cylinder's coverage area in (m^2).

To understand potential differences among treatments in N-NH_3 volatilization across all nine samplings, cumulative N-NH_3 was calculated for each treatment.

Total N emitted

Cumulative N loss from different treatments was calculated as N lost through N_2O emission and NH_3 volatilization. This was determined by linearly interpolating between sampling points, multiplying the mean N_2O emission and NH_3 volatilization for two successive sampling dates by the period between sampling, and adding that amount to the previous cumulative total (Dorich et al., 2020). The fraction of N emitted (N_2O emission and NH_3 volatilization) was obtained from the cumulative emission for each treatment. Nitrogen fraction (NF) was calculated using equation 4.

$$NF = \frac{MT - MC}{\text{Total N applied}} \quad \text{Eq. 4}$$

in which: NF is the N fraction emitted (N emitted as % of N applied); MT is the total N emitted (N_2O emission and NH_3 volatilization) per treatment; MC is the total N emitted from the control treatment; and Total N applied is the N applied as FDE (150 kg ha^{-1}).

Soil and plant parameters

Soil temperature was measured at 0.10 m depth. Soil composite samples (0.00–0.20 m layer) were taken to determine NO_3^- content (Cataldo et al., 1975) and gravimetric water content (GWC) by drying the soil samples at 105°C for 48 h or until constant weight was reached. Each soil composite sample was composed of three sub-samples. Soil samples were collected adjacent to the gas-flux chamber within the sampling area. Soil cylinders (100 cm^3 volume and 0.05 m in diameter) were also collected to determine soil bulk density (Blake, 1965).

Water-filled pore space (WFPS) was calculated using equation 5.

$$WFPS (\%) = \frac{\theta_g \times BD}{\left(1 - \frac{BD}{PD}\right)} \times 100 \quad \text{Eq. 5}$$

in which: θ_g (g/g) is the gravimetric water content; BD (g/cm^3) is the soil bulk density; and PD is the soil particle density, generally given as 2.65 Mg m^{-3} .

On day 30 DAA, the greenness index was measured (with SPAD 502 millivolt) as an average between four subsamples randomly chosen within each plot. Pasture aboveground was cut at 33 DAA in all plots. The greenness index was used as a non-destructive proxy for plant health and nitrogen status, providing insight into the effectiveness of each treatment in promoting pasture quality under drought conditions. The samples collected were dried in an oven at 40°C and weighed to obtain dry weight. Total N content (Modified Kjeldahl method, IRAM-SAGyP standard 29572:2018), the total C content (Modified Walkley & Black method, IRAM-SAGyP Standard 29571:2016), and the C/N ratio of the pasture were also determined and are shown in table 2.

Statistical analysis

Linear regression was used to estimate daily emission rates of N_2O based on three discrete flux measurements per gas, as previously described. These rates were then used to calculate cumulative emissions over time. To evaluate statistical differences between treatments in cumulative N_2O emissions, NH_3 volatilization, total N losses ($N_2O + NH_3$), pasture yield, and greenness index, data were subjected to analysis of variance (ANOVA) using the InfoStat software (Di Rienzo et al., 2020). Before ANOVA, assumptions of normality (Shapiro–Wilk test) and homoscedasticity (Levene’s test) were verified. Differences among means were tested using Tukey’s HSD test at a significance level of 0.05.

RESULTS

Emissions of N_2O were low throughout the experiment across all treatments, with a maximum of $18.40 \mu g m^{-2} h^{-1}$ of N- N_2O . The highest emissions were observed within the first DAA, with significant differences between the FDE and the rest of the treatments only on five sampling dates between days 4 and 12 DAA (Figure 1a). Soil temperature ranged from 17 to 27 °C, with an average of 20.5 °C (data not shown). The WFPS (water-filled pore space) ranged from 25 to 56 %, with an average of 42 % across the season, and did not differ among treatments. Values are therefore presented as the site-level mean in figure 1b. The seasonal mean WFPS was low (≈ 42 %), consistent with the drought conditions recorded. In all treatments with FDE application, the NO_3^- content was higher at the beginning of the experiment and decreased over time. Treatment with Zeo presented the highest NO_3^- values over time (Figure 1c).

Zeo or BC addition significantly reduced N_2O emissions by 63.1 and 63.8 %, respectively, compared with the FDE treatment. In contrast, the DCD addition did not present significant differences with the FDE treatment (Figure 2a). Regarding NH_3 volatilization, the BC treatment significantly reduced ($p < 0.05$) NH_3 volatilization by 55.5 % compared with the FDE treatment. The BC treatment showed NH_3 volatilization values close to those of the C treatment. In contrast, the Zeo and DCD treatments did not reduce NH_3 volatilization (Figure 2b). The BC treatment showed the highest inferred mitigation potential based on total N loss (the sum of N emitted as N_2O and NH_3) (Figure 2c).

Pasture yield was very low (close to $400 kg ha^{-1}$), and there were no significant differences between treatments (Figure 3a). However, there were significant ($p > 0.05$) treatment effects on greenness index with the following trend: BC (169.6 %) > FDE (145.3 %) $\approx$ DCD (138.4 %) $\approx$ Zeo (135.8 %) > C (100 %) treatment (Figure 3b).

DISCUSSION

Emissions of N_2O during the experiment were generally low, although short-lived peaks were observed shortly after the application of the treatments. These initial peaks were likely associated with the rapid increase in soil mineral nitrogen, particularly NO_3^- , following FDE application in all treatments (Figure 1c). Similar transient increases in N_2O emissions after the addition of organic amendments have been reported in previous studies (Roig et al., 2012; Masaka et al., 2014). For example, Li et al. (2015) found that N_2O fluxes consistently peaked within the first 24 h after FDE application across three consecutive seasons. Li et al. (2014) also observed this trend during winter. Comparable results were reported for other types of organic effluents, with sharp but short-lived increases in N_2O emissions after application (Sharpe and Harper, 2002; Luo et al., 2008).

The low cumulative N_2O emissions observed throughout the experiment can be largely attributed to the environmental conditions, particularly low soil moisture. Throughout the study period, WFPS did not exceed 60 % in any treatment, likely due to below-average rainfall. Under such conditions, the relative importance of denitrification as a source of N_2O diminishes, while nitrification becomes the dominant process (Linn and Doran, 1984). Since nitrification generally produces less N_2O than denitrification (Smith et al., 2003; Bateman and Baggs, 2005), this shift in dominant pathways likely contributed to the overall low emissions.

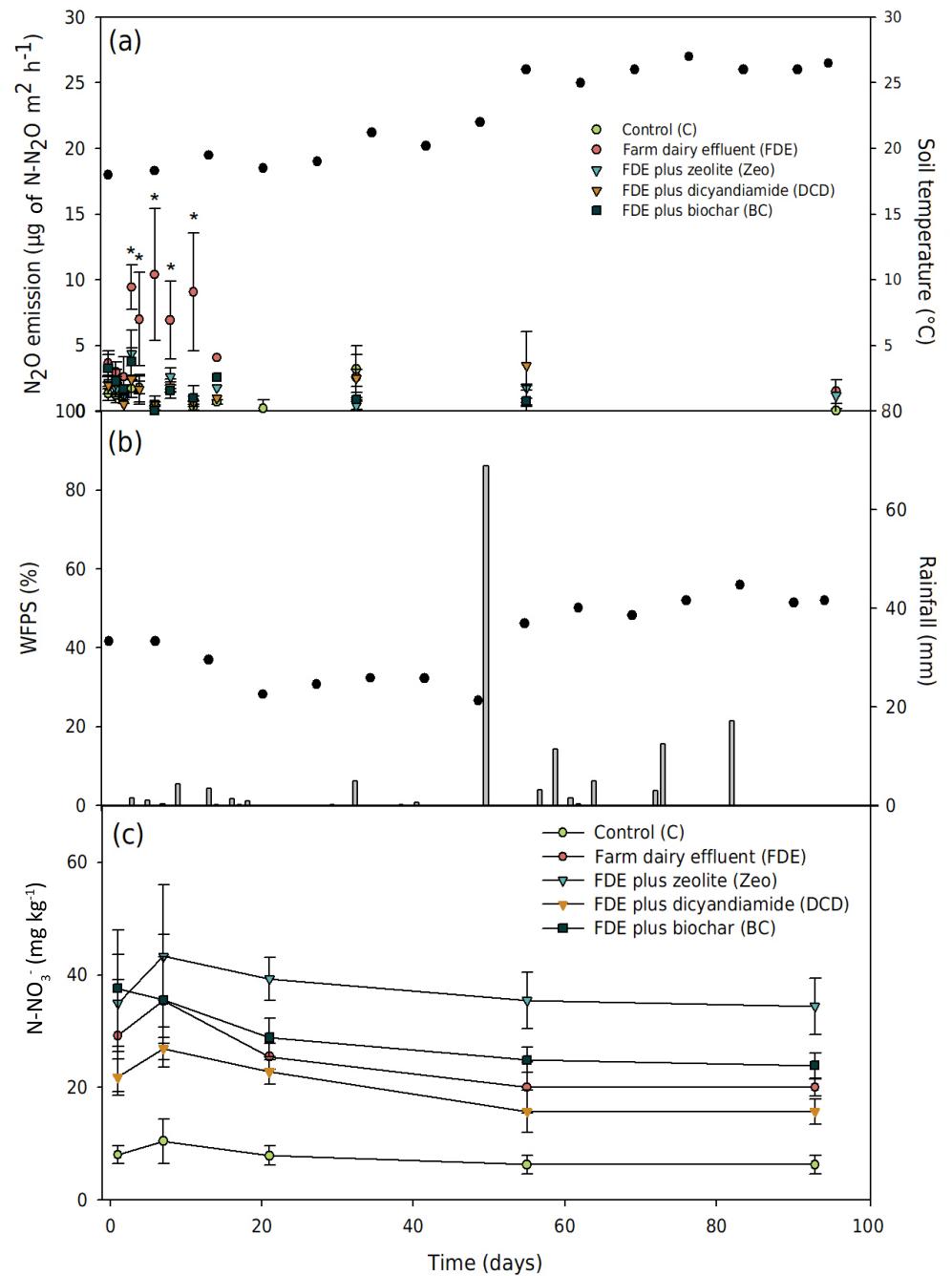


Figure 1. Evolution of N_2O emission (a), water-filled pore space (WFPS, $\circ$ -) and rainfall (b), and NO_3^- content (c) across treatments. Bars indicate standard errors. Asterisks (*) denote statistically significant differences between treatments for a given day ($p < 0.05$, ANOVA followed by Tukey's HSD test).

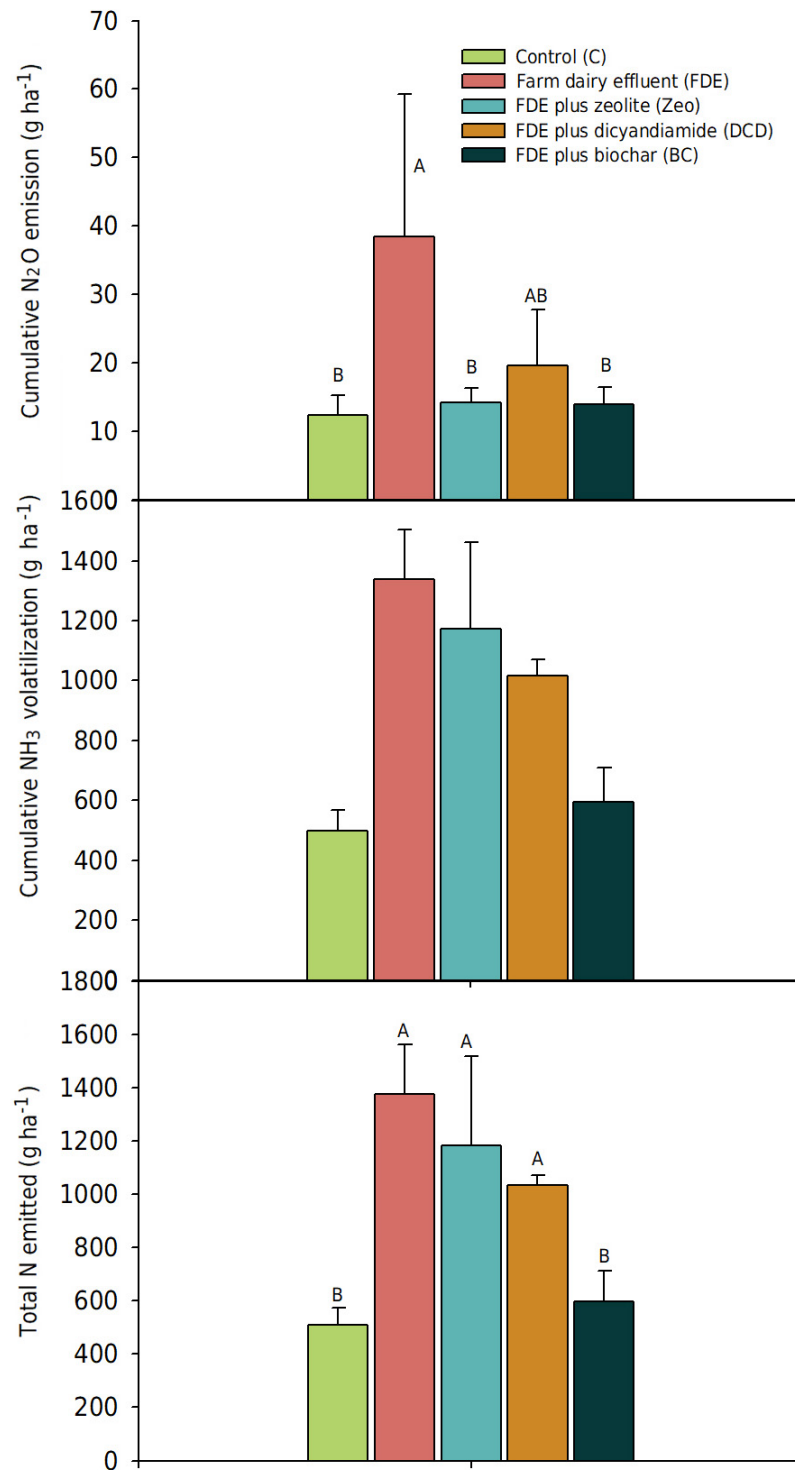


Figure 2. Cumulative N₂O emission (a), cumulative NH₃ volatilization (b), and total N lost to the atmosphere (c) from the control soil. Bars indicate standard errors. Different letters indicate statistically significant differences among treatments ($p < 0.05$, ANOVA followed by Tukey's HSD test).

Nevertheless, it is possible that the water added through FDE application temporarily created anaerobic microsites, promoting short-term denitrification and explaining the initial emission peaks (Siddique et al., 2012). Moreover, the average soil temperature during the study was 20.5 °C, which may have enhanced microbial activity. High temperatures can promote rapid oxygen consumption through microbial respiration, leading to the formation of anaerobic micropores and favoring denitrification (Li et al., 2000; Smith et al., 2003; Farquharson and Baldock, 2008). The combination of effluent-derived

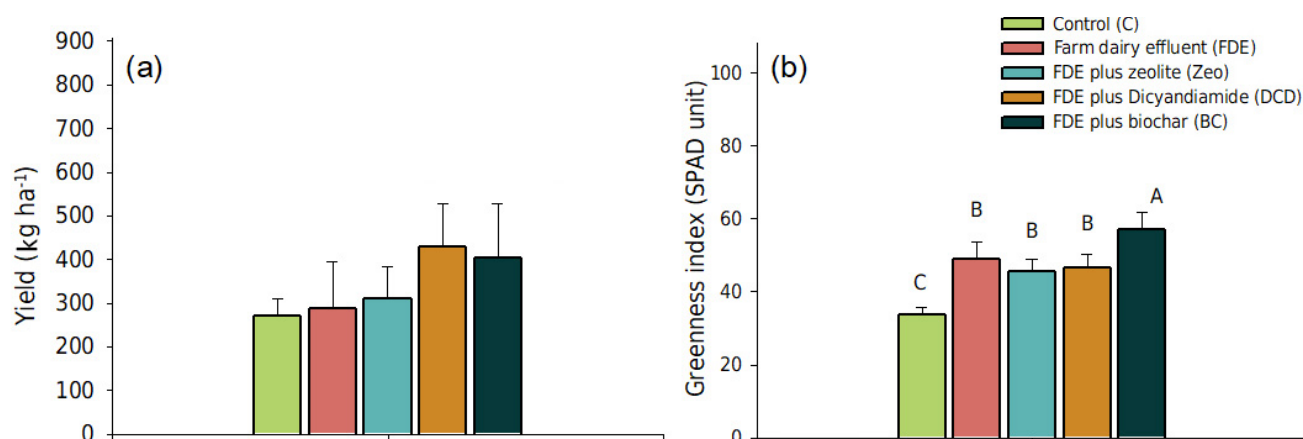


Figure 3. Pasture yield (a) and greenness index (b) from the control soil. Bars indicate standard errors. Different letters indicate statistically significant differences among treatments ($p < 0.05$, ANOVA followed by Tukey HSD test).

moisture and increased microbial activity could explain the brief spike in N_2O emissions observed immediately after application (Dalal et al., 2010). Importantly, in the medium and long term, a negative relationship was observed between soil temperature and WFPS, as elevated temperatures accelerated soil drying and reduced the formation of anaerobic zones. Consequently, the denitrification process induced by effluent application was likely short-lived.

No significant differences in cumulative N_2O emissions were observed between DCD and FDE treatment (Figure 2a). These results differ from those reported by many authors. Li et al. (2015) conducted a field experiment during spring, applying different organic fertilizers to the soil (Li et al., 2000). They observed that DCD reduced N_2O emissions by 24-69 %. Di et al. (2021) co-applied 10 kg of DCD ha⁻¹ with cow urine and observed a 65 % decrease in N_2O emission. Merino et al. (2002) also found that applying cattle manure plus 25 kg of DCD ha⁻¹ to the soil inhibited nitrification and reduced N_2O emissions by up to 60 %. A recent meta-analysis of 149 scientific publications found that DCD reduces N_2O emissions by 47 % without significantly affecting NH_3 volatilization (Tufail et al., 2022). Furthermore, Cosentino et al. (2024) observed a decrease of 50 % in N_2O emissions and an increase in crop yield when applying DCD together with FDE in an autumn-winter field experiment.

Under the conditions of the present study, with very dry soil and extremely hot weather (an average air temperature of 29 °C and 42 % WFPS), the DCD may have lost or reduced its efficiency and, therefore, its role as a mitigator. Soil moisture content strongly affects the effectiveness of DCD (Di and Cameron, 2006). Guo et al. (2022) investigated the effectiveness of four NIs (including DCD) in reducing N_2O emissions from N-fertilized soil. They concluded that this efficiency increased with increasing soil moisture. The effectiveness of DCD to mitigate N_2O emissions also depends on soil temperature. Below 15 °C, DCD has been reported to inhibit nitrification for up to 6 months, while above 15 °C, the efficacy decreases substantially (Kelliher et al., 2014). Furthermore, the DCD has low stability at high temperatures, which rapidly decreases its half-life (Kelliher et al., 2014).

Significant differences in cumulative N_2O emissions were observed between FDE and BC (Figure 2a). Pan et al. (2020) conducted a leachate test and observed that the considerable amount of surface functional groups and the large surface area of biochar effectively adsorbed and removed a significant amount of organic matter, facilitating the completion of the nitrification and denitrification reactions and resulting in lower N_2O emissions. Zhang et al. (2023) conducted a meta-analysis to analyze N_2O emissions from soil after biochar application. They found that, on average, adding biochar reduced N_2O emissions from soil by 16.2 %. Rittl et al. (2021) also applied different doses of biochar

in soils under various temperature conditions and observed that higher biochar rates were needed to mitigate N_2O emissions as soil temperature increased. For example, a dose of 2.5 Mg ha^{-1} was effective in reducing emissions in soils exposed to temperatures up to 40°C . These findings are consistent with our study, in which soil temperature averaged 20.5°C and biochar was applied at a lower rate (1.67 Mg ha^{-1}), yet still mitigated N_2O emissions.

Zeolite addition reduced N_2O emissions by 63.1 % (Figure 2a). This mitigation is consistent with zeolite high cation-exchange capacity and microporosity, which transiently retain NH_4^+ from the applied effluent/manure and modulate N release, damping short-lived mineral-N pulses that typically drive coupled nitrification–denitrification immediately after application. Consistent with this mechanism, multiple field and incubation studies report lower N_2O emissions with zeolite across N fertilizer sources (Zaman and Nguyen, 2010; Jumadi et al., 2020; Ali et al., 2022). When applied with pig slurry, zeolite decreased cumulative N_2O by 16.3 % and increased forage and cob yield in maize (Choi et al., 2020) and reduced cumulative N_2O by 15.6 and 31.5 % during king-grass and corn seasons, respectively, while improving NUE and N availability (Park et al., 2024). Under the dry, warm conditions of our study—low rainfall and prevailing drought—denitrification was likely constrained by limited anaerobic microsites (Dobbie and Smith, 2003), so the transient reduction in substrate availability and improved near-surface aeration associated with zeolite would further limit N_2O formation. The BC was the only treatment that reduced NH_3 volatilization (Figure 2b), reaching levels similar to those of the C treatment. Meirikhany et al. (2020) applied biochar to pig manure in storage and observed a reduction in NH_3 volatilization from 19 to 39 %. In contrast, the treatments with Zeo and DCD showed NH_3 volatilization values that were similar to those of FDE. The DCD is a NI, so it is not expected to reduce NH_3 losses from volatilization. In a meta-analysis conducted by Di et al. (2021) involving 89 studies worldwide, the authors concluded that, in most cases, the addition of NI increased NH_3 volatilization by 35.7 % compared with a fertilizer without NI, even under different soil conditions. Choi et al. (2020) observed that adding zeolite to pig manure reduced NH_3 volatilization by 20.1 %, while Park et al. (2024) found that zeolite application reduced cumulative NH_3 volatilization from swine manure by 16.7 and 24.4 % in the king-grass and corn growing seasons, respectively. Our results differ from those reported by these authors because we did not observe a decrease in NH_3 volatilization (Figure 2a). The balance between NH_3 and NH_4^+ in zeolites is influenced by water content; as water content decreases, NH_4^+ tends to convert to NH_3 . Lower water levels can lead to NH_4^+ being released as NH_3 rather than adsorbed by zeolite, thereby increasing NH_3 volatilization (Chen et al., 2018).

Pasture yield was low, and no significant difference between treatments was observed. However, the greenness index presented significant differences between treatments. The maximum value was observed in the BC treatment, intermediate values in the FDE, DCD, and Zeo treatments, and the lowest value in the C treatment. While the SPAD value is strongly related to plant N content, it is important to remember that it can also be affected by other factors that influence chlorophyll, such as Mg and S availability, plant diseases, and drought. Other researchers who have studied zeolite, biochar, and DCD applied to different agricultural residues reported nutritional improvements and pasture performance. Rehakova et al. (2004) reported that applying zeolite to soil modifies the rhizosphere and increases plant biomass. Niraula et al. (2021) observed that applying biochar saturated with dairy effluent to the soil improved the nutritional quality and growth of the pasture. Finally, Cosentino et al. (2024) observed increases in pasture yield after co-application of FDE plus DCD. In this experiment, the lack of rainfall may have affected pasture yield, interfering with the evaluation of the Zeo, BC, and DCD effects on yield.

Additionally, the application of zeolite generated a surface crust that delayed pasture growth. This crust may also have affected the pasture yield. This finding highlights the importance of avoiding doses that lead to crust formation on the soil surface.

CONCLUSIONS

In this study, during an exceptionally dry season, farm diary effluent (FDE) application with biochar (BC) was the most efficient mitigating strategy, showing the lowest loss of N to the environment and promoting higher quality in the pasture. In line with our hypothesis, the FDE + BC treatment reduced both N_2O and NH_3 losses and improved canopy greenness; by contrast, the hypothesis was not supported for zeolite, and no yield gains were detected.

Zeolite showed intermediate performance, mainly through reductions in N_2O , but it did not lessen NH_3 losses. The very low rainfall likely promoted surface crusting and limited the expression of zeolite's usual benefits; indeed, its high water-holding capacity can be counterproductive during extreme drought. The dicyandiamide (DCD) was ineffective at mitigating N losses (both N_2O and NH_3) under these conditions. Further multi-season studies under different environmental conditions are needed to expand recommendations on these mitigation substances.

DATA AVAILABILITY

After publication, the data will be available on demand to authors.

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