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Effectiveness and trade-offs of leonardite and rock powder additives (zeolite, bentonite, lava rock dust, diabase, and basalt) for ammonia and greenhouse gas mitigation during cattle slurry storage

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HIGHLIGHTS

- First comparative evaluation of rock powders (zeolite, bentonite, basalt, lava rock dust, diabase) and leonardite under controlled slurry-storage conditions with high-temporal-resolution gas monitoring.
- Cross-validation of ammonia measurement approaches using indophenol colorimetry and continuous Innova photoacoustic monitoring.
- Selected additives achieved substantial NH_3 mitigation, but performance differed markedly among additives and experiments.
- Results revealed pollution swapping, with NH_3 mitigation linked to increased greenhouse gas emissions.
- Excellent agreement was observed between indophenol colorimetry and continuous photoacoustic NH_3 monitoring analysis.

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ABSTRACT

Ammonia (NH_3) and greenhouse gas emissions from slurry storage represent major environmental challenges in livestock production. Additives such as humic substances and rock powders have been proposed as mitigation options. However, results regarding their effectiveness remain inconsistent, and their implications for nitrogen trade-offs between NH_3 and nitrous oxide (N_2O) emissions and total greenhouse gas emissions during slurry storage are poorly constrained. This study evaluated a humic substance (leonardite) and five rock powder additives (zeolite, bentonite, diabase, lava rock dust, and basalt) in controlled slurry storage. Additives were mixed with diluted untreated slurry at an application rate of 5% (w/w), and NH_3 emissions were quantified by continuous acid trapping, while, N_2O, methane (CH_4), and carbon dioxide (CO_2) were measured once daily during 10-min measurement periods, using the final 3 min for evaluation, over a 22-day storage period. Leonardite significantly reduced cumulative NH_3 emissions by 32%, followed by zeolite with a 29% reduction, whereas other additives did not significantly affect NH_3 emissions. However, ammonia reduction by leonardite was accompanied by a 20% increase in N_2O emissions, suggesting potential nitrogen trade-offs, while zeolite reduced NH_3 without increasing N_2O emissions. Diabase and lava rock dust significantly reduced CO_2 emissions by 20% and 14%, respectively. Across all treatments, CH_4 contributed most to total greenhouse gas emissions expressed as CO_2 equivalents and increased by up to 25–31% for some additives, offsetting potential environmental benefits from NH_3, even after inclusion of indirect N_2O emissions associated with NH_3 volatilisation. Consequently, no treatment resulted in a significant reduction in total greenhouse gas emissions expressed as CO_2

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equivalents. Overall, mitigation effects during slurry storage were limited and additive specific. These findings highlight the need for integrated, multi-gas assessments to ensure robust environmental benefits.

1. Introduction

Global demand for agricultural production, driven largely by rising consumption of animal-derived products, is projected to increase by nearly 70% by 2050 (Prado et al., 2020). This trend is expected to intensify livestock production, increasing the generation and on-farm management of manure, particularly cattle slurry (Amejo, 2024; Samad et al., 2025). Although slurry is an important, recycled, source of nitrogen (N), a substantial fraction is present as ammonium (NH_4^+), which drives ammonia (NH_3) emissions during storage and application to land (Sommer and Jensen, 1994). As a result, agriculture accounts for nearly 90% of global NH_3 emissions, contributing to air pollution and, following atmospheric deposition, to ecosystem acidification and eutrophication (Butterbach-Bahl et al., 2013; Covali et al., 2021; Ren et al., 2023; Taube et al., 2024).

In addition to NH_3, slurry management is a significant source of greenhouse gas (GHG) emissions, particularly methane (CH_4), a potent short-lived climate forcer, and nitrous oxide (N_2O), which has a high global warming potential and contributes to stratospheric ozone depletion (Gerber et al., 2013; Dennehy et al., 2017; Bittman, 2019; Kavanagh et al., 2021). Across storage and land application, cumulative gaseous losses can account for up to 40–50% of slurry-derived N and C, substantially reducing fertiliser value and increasing reliance on synthetic inputs (Petersen and Sørensen, 2008; Perazzolo et al., 2016). Mitigating these emissions is therefore central to improving nutrient-use efficiency and reducing environmental impacts in livestock production systems (Butterbach-Bahl et al., 2013; Kavanagh et al., 2021; Gioelli et al., 2022).

A range of mitigation strategies have been proposed to reduce slurry-related emissions (Ambrose et al., 2023). Among these, slurry acidification using sulfuric acid (H_2SO_4) is the most widely documented approach, as it lowers pH and stabilises N in the non-volatile NH_4^+ form (Fangueiro et al., 2015; Prado et al., 2020; Ambrose et al., 2023). Reported reductions of up to 75% for NH_3 and 61% for CH_4 indicate effectiveness under specific experimental conditions (Misselbrook et al., 2016; Prado et al., 2020; Emmerling et al., 2020; Regueiro et al., 2022). However, high implementation costs, occupational safety concerns, potential environmental risks, and incompatibility with organic farming systems limit its adoption (Loide et al., 2020; Prado et al., 2020; Overmeyer et al., 2021). These constraints have stimulated interest in simpler and more broadly applicable alternative mitigation strategies.

One alternative strategy involves amending slurry with adsorbent materials, including minerals, rock powders, biochar, and humic substances (Ndegwa et al., 2008; Swoboda et al., 2021; Cao et al., 2022). These materials typically exhibit high surface area and negative charge density, enabling adsorption or exchange of NH_4^+ and thereby reducing its availability for volatilisation as NH_3 (Ali et al., 2022; Cao et al., 2022).

In this context, the humic substance leonardite and rock powder additives such as zeolite, basalt, bentonite, and lava rock dust (Eifelgold®) have been proposed as NH_3 mitigation options (Swoboda et al., 2021; Ali et al., 2022). However, evidence for their effectiveness in reducing NH_3 emissions during slurry storage remains inconsistent and, in some cases, contradictory. This variability likely reflects the limited availability of controlled comparative experiments evaluating multiple additive performance under identical slurry storage, as most studies focus on soil incubation systems, post-application field trials, or solid and poultry manures, with relatively few addressing slurry storage directly (Swoboda et al., 2021; Ali et al., 2022; Cao et al., 2022; Khan et al., 2025). For example, Cao et al. (2022) reported NH_3 reductions of 32–64% following leonardite application in soil-only incubation studies, whereas Khan et al. (2025) observed negligible effects of leonardite on NH_3 emissions in soil–plant incubation experiments.

Similar inconsistencies have been reported for rock powder additives. For example, zeolite has been associated with increased NH_3 emissions of up to 50% under certain storage conditions (Amon et al., 1997) and reductions of approximately 50–60% under alternative storage or handling regimes (Lefcourt and Meisinger, 2001). A more recent study reported only moderate NH_3 mitigation by zeolite following soil application (Lee et al., 2025a), whereas another agricultural application study reported reductions of up to 92% for NH_3 and 65% for CH_4 (Ali et al., 2022). Similarly, lava rock dust reduced NH_3 emissions by 46% when applied to solid cattle manure under field conditions (Shah et al., 2012), while a controlled slurry storage study reported no mitigation (Swoboda et al., 2021). Comparable variability has been observed for basalt and other rock powders, with reported effects strongly dependent on manure type, application context, and experimental conditions (Witter and Kirchmann, 1989; Nuernberg et al., 2016; Swoboda et al., 2021; Curtis et al., 2023). Despite this uncertainty, commercial claims often promote emission reduction and nutrient benefits without validation under controlled slurry storage conditions (Swoboda et al., 2021).

Thus, the existing evidence base remains fragmented. Many studies focus exclusively on NH_3 emissions and do not consider downstream N transformations, limiting assessment of trade-offs to N_2O or the net effect on total GHG emissions (Prado et al., 2020; Cao et al., 2022). Moreover, mitigation outcomes for commonly studied materials such as zeolite and bentonite, remain strongly context dependent, as much of the available evidence is derived from soil-based experiments, land application of solid manures, or field studies with limited temporal resolution, rather than from controlled storage of raw slurry (Clem and Doehler, 1961; Nuernberg et al., 2016; Cao et al., 2022; Ali et al., 2022; Swoboda et al., 2021). In contrast, potentially promising materials such as basalt, characterised by high surface area and cation-exchange capacity (Navarre-Sitchler and Brantley, 2007; Nielsen and Fisk, 2010), and diabase, a chemically stable mafic rock with buffering properties, remain largely unexplored in the context of slurry emission mitigation (Murray, 2006; Borah et al., 2022). Consequently, one of the remaining knowledge gaps is the lack of controlled comparative studies evaluating humic and mineral additives simultaneously under identical slurry-storage conditions while assessing both NH_3 mitigation and potential greenhouse-gas trade-offs. Therefore, a systematic and comparative evaluation of rock powders and leonardite under controlled slurry storage conditions is required, including the simultaneous quantification of NH_3, N_2O, and CH_4 emissions, to assess mitigation performance, associated trade-offs between N loss pathways, and net GHG impact.

In parallel with uncertainties surrounding additive performance, accurate quantification of NH_3 emissions remains a methodological challenge. Laboratory studies commonly employ acid traps, but their temporal resolution depends on manual sampling frequency, and analyses are time-consuming (Ndegwa et al., 2009; Hafner et al., 2012; Lee et al., 2025b). Optical techniques such as photoacoustic multi-gas analysers enable continuous, high-frequency measurements of multiple gases, but require specialised incubation systems and automated sampling, limiting their widespread application. Direct comparisons between conventional acid trapping and high-resolution optical approaches remain scarce, particularly under identical experimental conditions, limiting methodological validation and contributing uncertainty to comparisons of reported emission dynamics.

Against this background, the objectives of this study are to: (i) evaluate the effectiveness of the humic substance leonardite and the rock-based additives zeolite, bentonite, diabase, lava rock dust, and

basalt in reducing NH_3 emissions from slurry; (ii) assess trade-offs between NH_3 and N_2O emissions; (iii) characterise total greenhouse gas dynamics (N_2O, CH_4, and CO_2) and their contribution to global warming potential; and (iv) assess the consistency of NH_3 emission estimates derived from conventional acid trapping and high-frequency optical measurement techniques.

2. Materials and methods

2.1. Slurry and additives

Cattle slurry was obtained from the organic research farm Gladbacherhof (Justus Liebig University Giessen, Hesse, Germany), which operates under Bioland and Naturland organic certification. The dairy herd was managed under the high-input feeding regime at Gladbacherhof, as described by Eisert et al. (2025). The slurry consisting of urine, faeces, and residual bedding material, was representative of organic dairy production systems in central Germany. The barn was equipped with a robotic manure removal system. Slurry remained in the barn collection system for approximately 1–2 weeks, depending on the amount accumulated. Prior to sampling, slurry was homogenised in the barn using a mechanical mixer and subsequently pumped through the transfer system towards the slurry storage tank. Immediately after mixing, subsamples were collected from the transfer channel, before the slurry entered the storage tank, into two 100 L polyethylene barrels and stored at 4 °C for 24 h prior to use. On the day of experimentation, a representative 500 mL aliquot of untreated slurry was taken and submitted to an accredited external laboratory (Hessisches Landeslabor, Standort Kassel, 34123 Hesse, Germany) for analysis of baseline physicochemical properties (Table 1; control slurry CS1 and CS2). Total nitrogen (TN) was determined by combustion analysis, ammonium-N by colorimetric analysis using automated flow techniques, dry matter (DM) after oven drying to constant mass, and organic matter (OM) as loss on ignition.

To evaluate slurry amendments, five mineral additives (zeolite, bentonite, lava rock dust, diabase, and basalt) were sourced from suppliers in southern Germany (Table S1). Leonardite, an oxidised lignite-based humic amendment, was obtained from a regional supplier. All additives were stored at room temperature, in the dark at the Institute of Animal Nutrition and Feed Science, Justus Liebig University Giessen, until use. Prior to application, additives were sieved to <2 mm to ensure an outbalanced particle size.

2.2. Experimental setup and design

The experiments were conducted under controlled laboratory conditions in an air-conditioned room at 20 °C at the Institute of Animal Nutrition and Feed Science, Justus Liebig University Giessen, Germany. To account for variability in gaseous emissions, each treatment was established with three independent replicates (n = 3).

Due to laboratory space constraints, the full experimental design was implemented in two consecutive slurry storage experiments using the same batch of slurry and an identical experimental protocol. The first experiment comprised Slurry + Leonardite (SL), Slurry + Zeolite (SZ), and Slurry + Bentonite (SB). The second experiment comprised Slurry + Diabase (SD), Slurry + Lava rock dust (SLRD), and Slurry + Basalt (SBT). In both experiments, untreated slurry served as a common control (CS) (see Fig. 1). Accordingly, treatment effects were evaluated relative to the control within each experiment, and direct statistical comparisons between the two experimental runs were not performed. Potential temporal effects associated with the sequential execution of the experiments were accounted for in the statistical analysis (Section 2.4).

Under farm conditions, rock powder additives are commonly incorporated into slurry by stirring using tractor-driven rotors, promoting uniform distribution. To create a laboratory analogue, slurry was first homogenised in 100 L barrels and then 20 L were transferred into a dedicated mixing barrel for each treatment. This amount provided sufficient material to prepare three replicate storage barrels per treatment and subsample for physicochemical analysis. Although suppliers typically recommend additive rates of 2–3% (w/w), previous studies indicate that the optimal application rate, particularly for leonardite, is not well established and may exceed these recommendations (Cao et al., 2022; Khan et al., 2025). Therefore, an application rate of 5% (w/w, fresh-mass basis) was used in this study. The pre-sieved additives were evenly distributed over the slurry surface before mixing. Each mixture was blended for 2 min using a hand mixer (IKARI ILB 63-2B14; IKARI Motors GmbH, Germany) to obtain consistent incorporation. Immediately after mixing, a 500 mL subsample was collected and sent to an external laboratory for physicochemical analysis (Hessisches Landeslabor, Standort Kassel, 34123 Hesse, Germany; Table 1).

The remaining slurry was used to prepare the replicates: 6 L of the treated slurry were transferred into each of three airtight 12 L polyethylene storage barrels (Speidel Tank-und Behälterbau GmbH, 72131 Ofterdingen, Germany). This procedure, including homogenization, mixing, subsampling, and filling of the three replicate storage barrels, was repeated identically for each treatment in both experiments. To reduce adhesion of solids to the container walls and facilitate residues recovery, the inner surfaces were lined with 1-mm polytetrafluoroethylene sheets (PTFE, Carl Roth GmbH, Karlsruhe, Germany) and coated with a thin layer of PTFE-based release spray prior to filling. The barrels were then sealed promptly to ensure gas-tight storage, and NH_3 emissions were trapped continuously, while GHG were measured once daily to quantify gaseous losses during storage. Crust formation was monitored by visual inspection of the slurry surface following each daily

Table 1
Physicochemical properties of slurry and additive treatments used in the two controlled storage experiments. The parameters measured include pH, total nitrogen (TN, g/kg), ammonium (NH_4^+-N, g/kg), organic matter (OM, %) and dry matter (DM, %) and additives mixing ratio (AMR, %). Additives were applied at an application rate of 5% (w/w) relative to slurry fresh weight. Experiment 1 included control slurry (CS1), Slurry with leonardite (SL), zeolite (SZ), and bentonite (SB), while experiment 2 included control slurry (CS2), diabase (SD), lava rock dust (LRD), and basalt (SBT).

Experiment	Treatment	pH	Total nitrogen (TN, g/kg)	Ammonium (NH_4^+-N, g/kg)	Organic matter (OM, %)	Dry matter (DM, %)	Moisture content (MC, %)	Additives mixing ratio (AMR, %)
1	Control slurry (CS1)	6.60	2.23	0.85	36.71	8.58	91.42	-
	Slurry + Leonardite (SL)	6.41	2.35	0.81	36.37	11.55	88.45	5
	Slurry + Zeolite (SZ)	6.50	2.16	0.82	25.71	11.37	88.63	5
	Slurry + Bentonite (SB)	6.71	2.14	0.82	25.52	12.30	87.70	5
2	Control slurry (CS2)	6.80	2.59	1.10	35.72	8.26	91.74	-
	Slurry + Diabase (SD)	6.81	2.49	1.06	24.41	12.22	87.78	5
	Slurry + Lava rock dust (SLRD)	6.90	2.47	1.03	22.19	12.25	87.75	5
	Slurry + Basalt (SBT)	7.00	2.48	1.03	24.58	12.14	87.86	5

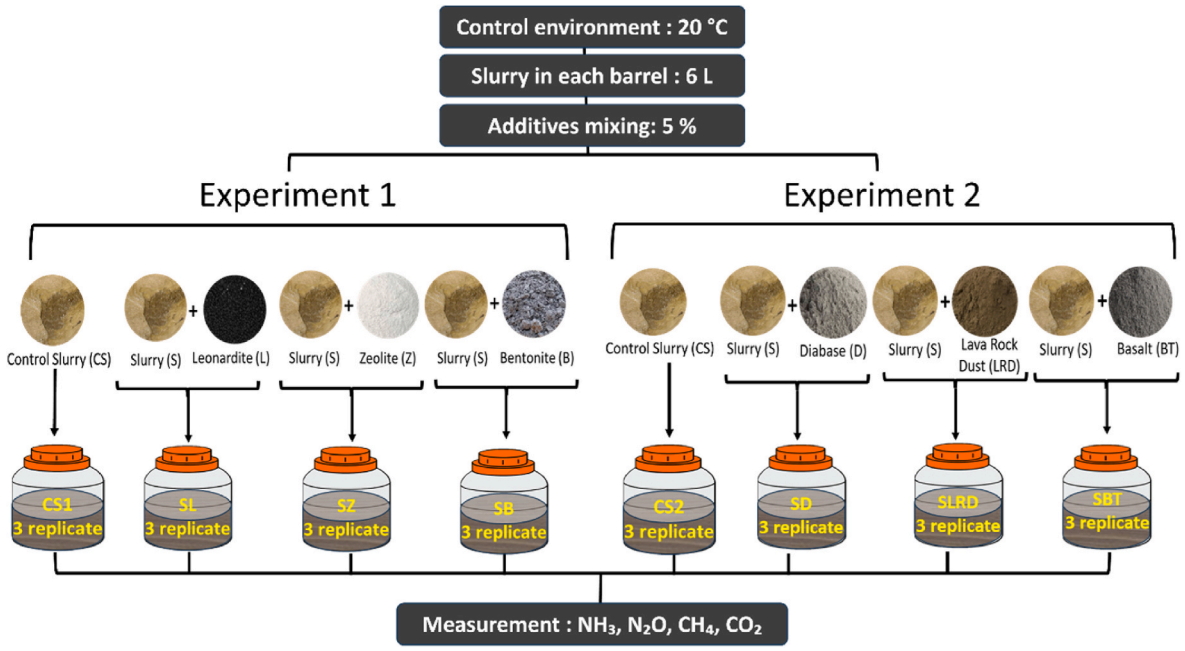


Fig. 1. Experimental design of the controlled slurry storage trials. Two consecutive experiments were conducted under identical conditions (20 °C), each including an untreated control (CS) and three additive treatments (n = 3), using the same slurry source and experimental protocol. Additives were applied at 5% (w/w), and 6 L of slurry were stored per barrel for 22-days. NH_3 emissions were quantified by continuous acid trapping, while N_2O, CH_4, and CO_2 were measured once daily.

gas measurement. For this purpose, the barrels were briefly opened and immediately closed again to minimize disturbance. This procedure was applied consistently across all treatments.

2.3. Measurement of trace gas fluxes (NH_3, N_2O, CH_4, and CO_2)

Ammonia and greenhouse gas emissions were quantified using a flow-through chamber system with continuous NH_3 trapping and intermittent gas concentration measurements. Each 12 L slurry storage barrel functioned as an airtight chamber (6 L headspace) with air inlet and outlet. A continuous airflow of 2 L min^{-1} was maintained through each barrel using an individual diaphragm pump, ensuring that barrels were not interconnected, under near-ambient pressure. Incoming air passed over the slurry surface and exited through the outlet line. The system was operated as a dynamic flow-through chamber, and gas fluxes were derived from the difference between outlet and inlet concentrations (ambient air) combined with airflow rate and chamber surface area. These conditions correspond to a residence time of approximately 3 min, suggesting that steady-state conditions were reached within each 10 min measurement period. All gas lines consisted of 6 mm PTFE tubing to ensure inert and airtight gas transfer.

The outlet line of each barrel was equipped with a T-junction and ball valves upstream of the washing bottle with only one line being active at a time and one line directing the air flow through washing bottles and the other line directing the air flow to a multi-gas analyser. The outlet line connected to the washing bottle was active for the whole day, except for 10 min during which it was closed and the line connected to the multi-gas analyser was opened. Thus, volatilized NH_3 was captured for 23 h and 50 min per day by directing outlet air from each barrel through a borosilicate gas-washing bottle (250 mL; Model EU0002175, ALWSCI Technologies, Karlsruhe, Germany) containing 150 mL of 0.1 M H_2SO_4, where NH_3 was quantitatively retained as NH_4^+ (Bolleter et al., 1961). The acid-traps were replaced daily during the first week, when NH_3 emissions were expected to be highest, and every second day thereafter. Collected trap solutions were stored at −20 °C until analysis. Prior to measurement, samples with high NH_4^+ concentrations were diluted with fresh 0.1 M H_2SO_4. Ammonium concentrations were determined colorimetrically using the Berthelot reaction, forming indophenol blue, and analysed with a continuous flow analyser (SEAL AA3 HR, SEAL Analytical, Southampton, United Kingdom, Bolleter et al., 1961) following method D-102-93 (Rev. 8) in accordance with DIN 38406.

Greenhouse gas (N_2O, CH_4, and CO_2) as well as ammonia (NH_3) concentrations were measured using a photoacoustic multi-gas analyser (INNOVA 1412a; LumaSense Technologies A/S, Ballerup, Denmark; Fig. 2). For these measurements, the outlet line of each barrel was equipped with a gas-tight T-connector upstream of the acid trap. During daily measurements, the acid-trap line was temporarily closed, and outlet air was diverted to the analyser. Each barrel was connected individually to the analyser for a 10 min measurement period. To minimize transient effects associated with switching and line exchange, only the final 3 min of each measurement period were used for data evaluation. Between successive barrels, ambient air was measured for 5 min to re-establish baseline concentrations. The analyser was operated under standard calibration conditions (20 °C, 101.33 kPa) with automatic flushing and a 5 s integration time. Gas concentrations were measured once daily for 22-days following slurry–additive mixing. The 22-day storage period was chosen to assess the short-term mitigation potential of the additives, covering the initial emission response to slurry–additive mixing and subsequent changes as surface crusts began to develop.

2.4. Data processing and statistical analysis

Trace gas fluxes of NH_3, N_2O, CH_4, and CO_2 were calculated in Microsoft Excel (Office, 2019; Microsoft, Seattle, WA, USA). Gas concentrations (ppm) measured by the INNOVA were corrected for background concentrations recorded between samples. Fluxes were derived using a dynamic flow-through chamber approach based on the difference between outlet and inlet concentrations, the known airflow rate, and the chamber surface area, following established methods (Pape et al., 2009; Khan et al., 2025). Concentrations were converted to mass-based fluxes by accounting for the molecular weight of each gas, the chamber airflow rate, the molar volume of air under standard conditions, and the effective surface area of the slurry exposed within the chamber. Fluxes were expressed as mg m^{-2} h^{-1}, with daily emissions calculated as 24-fold hourly fluxes (mg m^{-2} day^{-1}) and cumulative

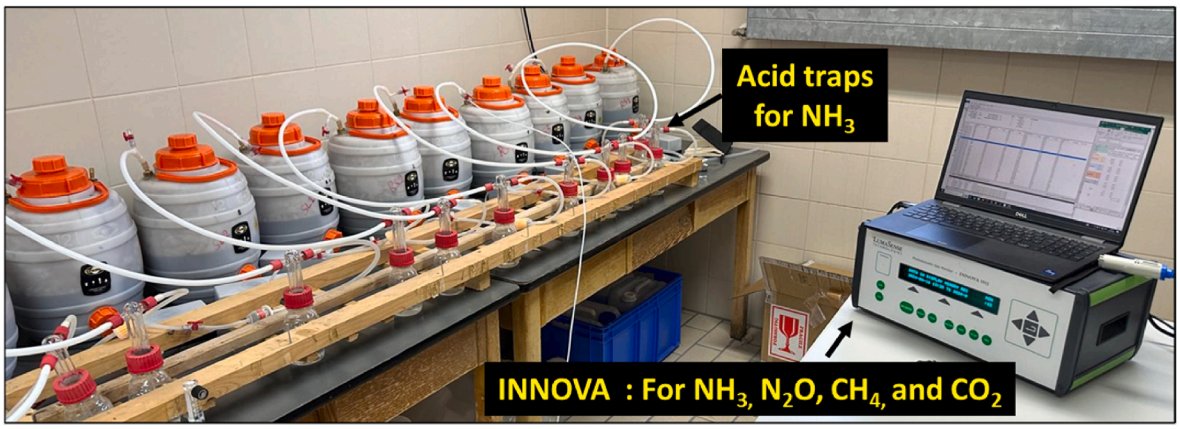


Fig. 2. Experimental setup of the dynamic chamber system used for slurry storage experiments, showing sealed slurry barrels connected in parallel to acid traps for NH_3 capture and to an INNOVA photoacoustic multi-gas analyser for high-temporal-resolution-measurement of NH_3, N_2O, CH_4, and CO_2.

emissions obtained by summing daily values over the measurement period (g m^{-2}).

Indirect N_2O emissions associated with NH_3 volatilisation were estimated according to the IPCC Guidelines for National Greenhouse Gas Inventories (Hou et al., 2015; Hergoualc'h et al., 2019). Because cumulative NH_3–N emissions were measured directly using the acid-trap method, indirect N_2O–N emissions were calculated by applying the default emission factor $EF_4 = 0.01$ kg N_2O–N kg^{-1} NH_3–N volatilized. Indirect N_2O–N was converted to N_2O using the molecular weight ratio of 44/28 and then converted to CO_2 equivalents using the same GWP_{100} factor applied to directly measured N_2O. The resulting values were included in the calculation of total greenhouse gas emissions expressed as CO_2 equivalents.

Total greenhouse gas emissions were expressed as CO_2 equivalents (CO_2-eq) using 100-year global warming potential (GWP_{100}) factors of 27 for CH_4 (non-fossil) and 273 for N_2O (Masson-Delmotte et al., 2021). Estimated indirect N_2O emissions were used only for the GHG balance and were not included in the treatment-effect or elastic net analyses, which were based exclusively on directly measured gaseous emissions.

All statistical analyses were performed separately for experiment 1 and experiment 2. Although both experiments used slurry from the same barn storage and followed an identical protocol, variation in physicochemical properties (e.g. total nitrogen, NH_4^+–N, dry matter content, and pH) may arise from handling, heterogeneity, and temporal dynamics (Khan et al., 2025). Because cumulative gaseous emissions are sensitive to these properties, absolute emission values were not compared across experiments. Accordingly, treatment effects were evaluated within each experiment relative to its respective untreated control, to isolate additive effects from baseline variability.

For each experiment, treatment effects on cumulative emissions were assessed using one-way analysis of variance (ANOVA), followed by Tukey's honest significant difference (HSD) test for pairwise comparisons where appropriate. Model residuals were evaluated for normality (Shapiro–Wilk test) and homogeneity of variance (Levene's test). Given the small sample size ($n = 3$), these diagnostics were interpreted cautiously, and ANOVA was considered robust to moderate deviations from normality. Statistical significance was defined at $p < 0.05$.

Subsequently, an elastic net regression model was fitted on the combined dataset to identify slurry parameters associated with cumulative emissions of individual gases, including experiment as a categorical predictor to account for between-experiment differences. Elastic net regression combines L1 (lasso) and L2 (ridge) penalties, allowing variable selection in the presence of multicollinearity and limited sample size. Predictor variables were standardised, and regularisation parameters were selected by cross-validation. These analyses were used to identify statistical associations rather than infer causal relationships. All statistical analyses, including normality testing, homogeneity testing, ANOVA, Tukey's HSD test, and elastic net regression, were performed in R (version 4.4.2; R Core Team, Vienna, Austria). Elastic net regression was implemented using the glmnet package (Friedman et al., 2010). Graphical outputs and summary figures were produced using OriginPro 2020 (OriginLab Corporation, Northampton, MA, USA).

3. Results

3.1. Influence of additives on physicochemical properties of slurries

The physicochemical properties of the control and amended slurries from both experiments are summarised in Table 1. As expected, the addition of solid additives (5% w/w) increased dry matter content and reduced moisture content across all treatments. In experiment 1, the control slurry (CS1) had a pH of 6.60, TN content of 2.23 g kg^{-1}, an ammonium-N concentration of 0.85 g kg^{-1}, and an OM content of 36.71%. Among the additives, leonardite reduced slurry pH to 6.41 and slightly increased TN to 2.35 g kg^{-1}, whereas the zeolite and bentonite treatments were associated with lower OM contents of approximately 25.60%.

In experiment 2, the control slurry (CS2) had a TN concentration of 2.59 g kg^{-1}, an NH_4^+-N concentration of 1.10 g kg^{-1}, and a pH of 6.80. Amending slurry with diabase, lava rock dust, or basalt resulted in a slight increase in pH (6.81 to 7.00), while OM content was lower compared with CS2 (22.19–24.58% vs 35.72%). Total nitrogen and ammonium-N concentrations were marginally lower in amended slurries than in the CS2, although differences were small.

3.2. Influence of additives on ammonia emission

As NH_3 emissions were quantified using both acid traps and photoacoustic measurements, results derived from the acid-trap method are presented first, followed by photoacoustic INNOVA-based NH_3 concentration data (Section 3.3), and their comparison (section 3.7).

Across both experiments (Fig. 3), NH_3 emissions followed a consistent temporal pattern. Ammonia emissions remained low during the first week after slurry–additive mixing (days D1–D7). After this period, the values measured on D9, D11, D13, and D15 were higher. In experiment 1, a visible surface crust developed around D13 (Fig. 3a), and in experiment 2 around D15 (Fig. 3b). Within each experiment, the timing and persistence of visible surface crust formation were similar across all amendment treatments, with no clear amendment-specific differences observed. Following crust formation, emissions remained at a high level but dropped to a level similar to D1 to D7 on days 22 and 20 (experiments 1 and 2, respectively).

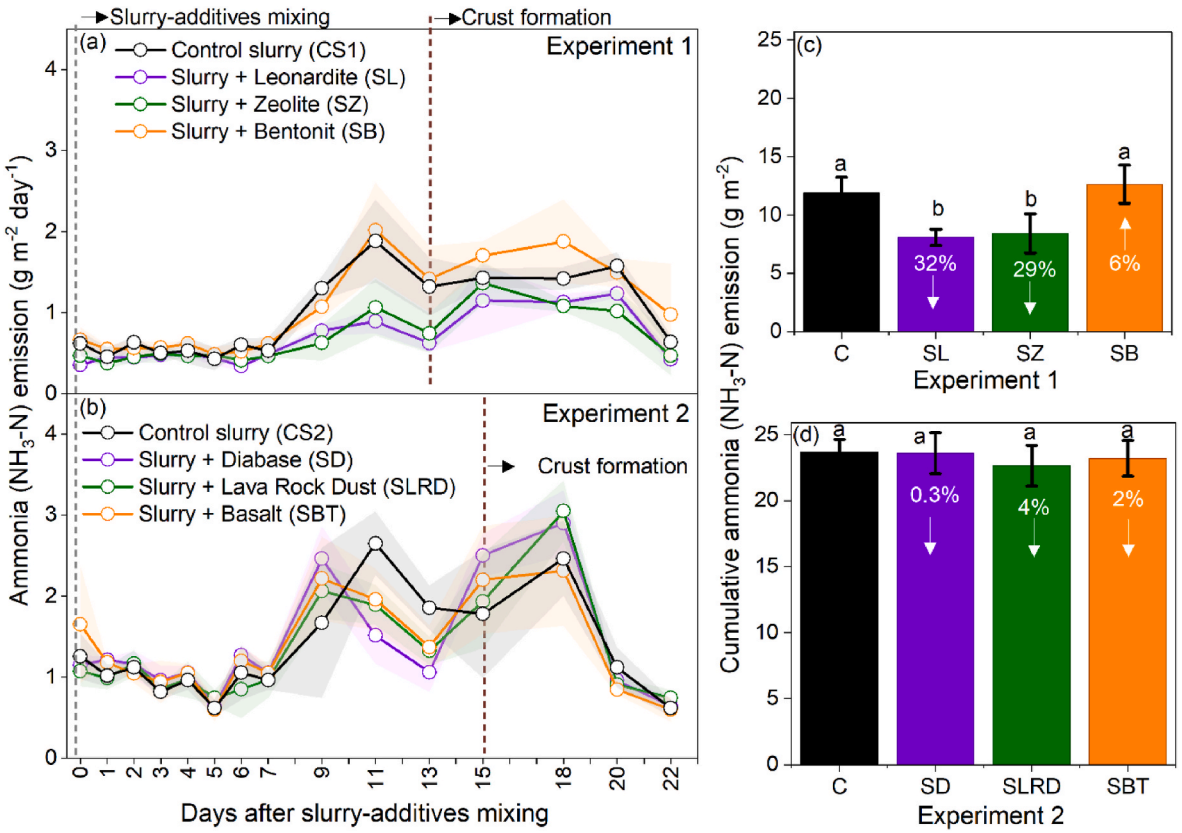


Fig. 3. Daily ammonia fluxes (NH_3-N) of (a, b) and cumulative emissions (c, d) from experiment 1 and 2, respectively, determined using acid traps. Treatments control slurry (CS1), slurry + leonardite (SL), slurry + zeolite (SZ) and slurry + bentonite (SB) in experiment 1, and control slurry (CS2) slurry + diabase (SD), slurry + lava rock dust (SLRD) and slurry + basalt (SBT) in experiment 2 are shown in black, violet, green and orange colour, respectively. Gray dotted lines mark the day of additive–slurry mixing, and red dotted lines indicate the onset of surface crust formation. Arrows with percentage values on bar graphs indicate the magnitude and direction of change in emissions relative to the control slurry. Mean daily fluxes (g m^{-2} day^{-1}) are shown as coloured symbols with shaded standard deviation (SD) bands, whereas cumulative emissions (g m^{-2}) are shown as bars with whiskers. Shaded bands and whiskers represent ± SD (n = 3). Bars sharing the same lowercase letter are not statistically different ($p > 0.05$), whereas bars with different letters differ statistically ($p < 0.05$) according to Tukey's HSD test. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

Cumulative NH_3 emissions were analysed separately for each experiment relative to the corresponding control slurry. In experiment 1 (Fig. 3c), cumulative NH_3 emissions from slurry amended with leonardite and zeolite were reduced by 32% and 29%, respectively, compared with the control slurry (CS1), and these reductions were statistically significant ($p < 0.05$). In contrast, cumulative NH_3 emissions from bentonite-amended slurry were 6% higher than CS1, although this difference was not statistically significant ($p > 0.05$).

In experiment 2 (Fig. 3d), cumulative NH_3 emissions from slurry amended with diabase, lava rock dust, or basalt differed by <4% from the control slurry (CS2), and none of these differences were statistically significant.

3.3. *Effect of additives on trace gas fluxes (NH_3, N_2O, CH_4, and CO_2)*

Fluxes of N_2O, CH_4, and CO_2, together with NH_3 concentrations measured by the photoacoustic INNOVA analyser across both experiments are presented (Figs. 4 and 5). In contrast to Section 3.2, where NH_3 emissions were quantified using acid trap measurements, the NH_3 data presented here represent high-temporal-resolution concentration measurements. All gases exhibited similar temporal patterns across the treatments of the experiment, although the timing and magnitude of fluxes differed between experiments. In experiment 1 (Fig. 4a–d), fluxes of all gases were low immediately after slurry–additive mixing (D1), followed by modest day-to-day variability and generally stable emissions until approximately D13, when surface crust formation occurred. After crust formation, NH_3 fluxes declined gradually, whereas N_2O, CH_4, and CO_2 fluxes continued to increase. Across all treatments, NH_3 fluxes ranged from 8.9 to 22.3 mg m^{-2} h^{-1}, N_2O from 1.0 to 2.1 mg m^{-2} h^{-1}, CH_4 from 13.8 to 180.6 mg m^{-2} h^{-1}, and CO_2 from 557.1 to 1811.2 mg m^{-2} h^{-1} (Fig. 4a–d).

Cumulative emissions in experiment 1 (Fig. 4e–h) were analysed relative to the control slurry (CS1). Cumulative NH_3 emissions were statistically lower in slurry amended with leonardite and zeolite than in CS1 ($p < 0.05$), with reductions of 17% and 22% (Fig. 4e), respectively, whereas slurry amended with bentonite did not differ statistically from CS1. For N_2O, cumulative emissions were statistically higher in slurry amended with leonardite than in CS1 ($p < 0.05$), with an increase of 20% (Fig. 4f), while zeolite and bentonite amended slurry did not differ statistically from CS1 (≤4%). Cumulative CH_4 emissions were statistically higher for slurry amended with bentonite than in CS1 ($p < 0.05$), with an increase of 25% (Fig. 4g), while leonardite amended slurry had a non-significant mean increase of approximately 31%. Cumulative CO_2 emissions did not differ statistically between amended treatments and the CS1, although mean emissions were lower for amended slurry with leonardite (−8%, Fig. 4h) and zeolite (−11%) and slightly higher for bentonite.

In experiment 2 (Fig. 5a–d), NH_3 fluxes were highest immediately after mixing, remained low until approximately D9, increased steadily until crust formation on D15, and subsequently declined. In contrast, N_2O, CH_4, and CO_2 fluxes decreased immediately after mixing, increased during the first few days, and then declined again until crust formation. Following crust development, small increases in N_2O and CH_4 fluxes were observed, while CO_2 fluxes remained relatively stable. Across all

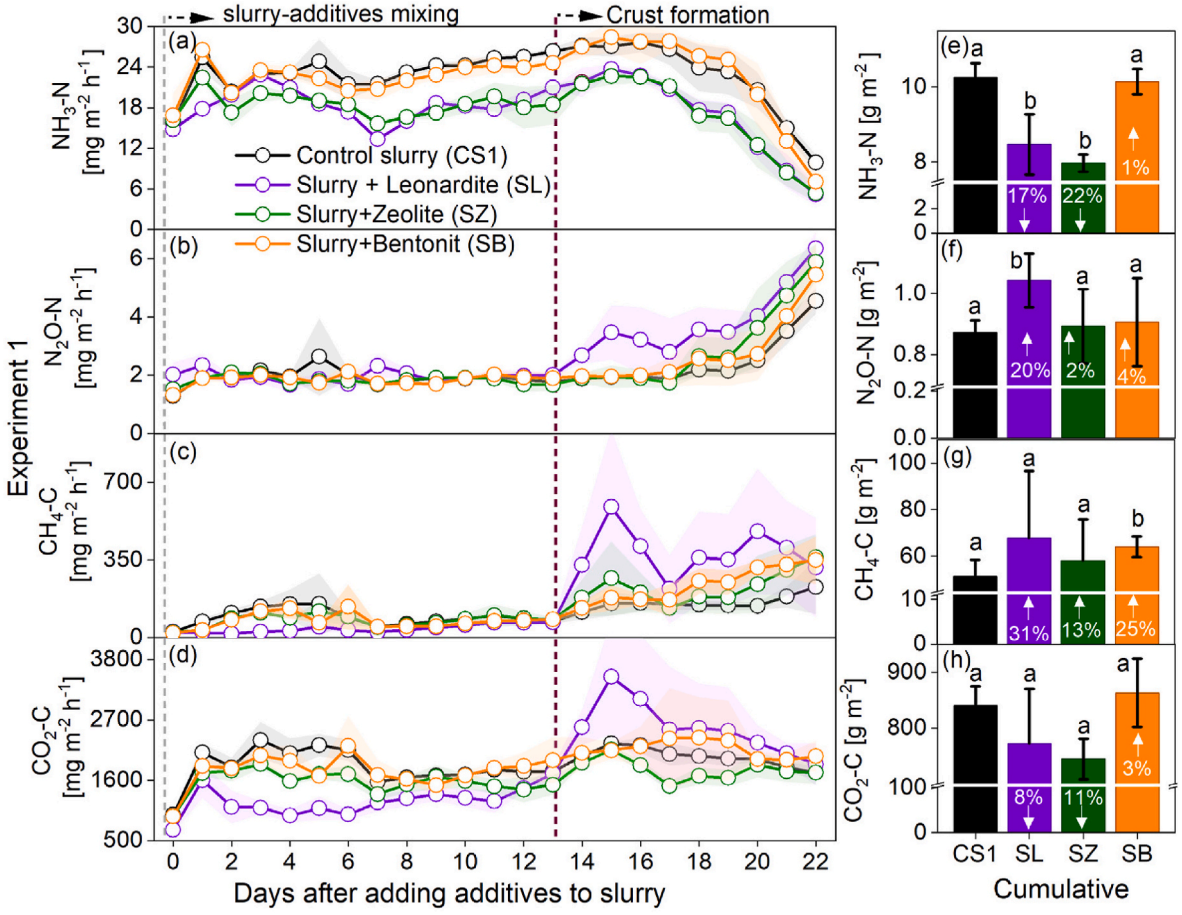


Fig. 4. Daily fluxes (a, b, c, d) and cumulative emissions (e, f, g, h) of ammonia (NH_3-N), nitrous oxide (N_2O-N), methane (CH_4-C), and carbon dioxide (CO_2-C) from experiment 1. Treatments control slurry (CS1), slurry + leonardite (SL), slurry + zeolite (SZ) and slurry + bentonite (SB) are shown in black, violet, green and orange colour, respectively. Gray dotted lines mark the day of additive–slurry mixing, and red dotted lines indicate the onset of surface crust formation. Arrows with percentage values on bar graphs indicate the magnitude and direction of change in emissions relative to the control. Mean daily fluxes (g m^{-2} day^{-1}) are shown as coloured symbols with shaded standard deviation (SD) bands, whereas cumulative emissions (g m^{-2}) are shown as bars with whiskers. Shaded bands and whiskers represent ± SD (n = 3). Bars sharing the same lowercase letter are not statistically different ($p > 0.05$), whereas bars with different letters differ statistically ($p < 0.05$) according to Tukey's HSD test. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

treatments, NH_3 fluxes ranged from 29.0 to 45.2 mg m^{-2} h^{-1}, N_2O from 1.4 to 2.1 mg m^{-2} h^{-1}, CH_4 from 17.7 to 179.2 mg m^{-2} h^{-1}, and CO_2 from 735 to 2339 mg m^{-2} h^{-1} (Fig. 5a–d).

Cumulative NH_3 emissions in experiment 2 (Fig. 5e–h) differed only slightly relative to the control slurry (CS2). Slurry amended with diabase had statistically higher NH_3 emissions than CS2 ($p < 0.05$), with an increase of 6%, whereas slurry amended with lava rock dust and basalt had non-significant increases (+3% and +2%, respectively; Fig. 5e). For N_2O emissions, slurry amended with diabase had a small, non-significant increase of 5%, whereas slurry amended with lava rock dust and slurry amended with basalt exhibited non-significant decreases (−6% and −3%, respectively; Fig. 5f), relative to CS2. Cumulative CH_4 emissions were statistically higher in slurry amended with basalt than in CS2 with an increase of 16% (Fig. 5g), whereas diabase and lava rock dust amended slurry resulted in non-significant decreases (−2% and −4%, respectively), relative to CS2. In contrast, cumulative CO_2 emissions were statistically lower in slurry amended with diabase and lava rock dust than in CS2, with a reduction of 20% and 14%, respectively (Fig. 5h), while emissions in slurry amended with basalt did not differ statistically from CS2.

3.4. Nitrogen-related trade-offs between NH_3 and direct N_2O emissions

In experiment 1, slurry amended with leonardite reduced cumulative NH_3 emissions but was accompanied by a significant increase in direct N_2O (Fig. 4f). In contrast, zeolite reduced NH_3 emissions without increasing direct N_2O emissions, whereas bentonite had no significant effect on either gas. In experiment 2, none of the tested additives reduced cumulative NH_3 emissions, and no statistically significant changes in direct N_2O emission were observed (Fig. 5f).

3.5. Total greenhouse gas emissions (direct N_2O, indirect N_2O and CH_4) expressed as CO_2 equivalents (CO_2-eq)

Total GHG emissions, expressed as CO_2 equivalents (Table 2), exhibited no statistical treatment effects in experiment 1 and only one significant effect in experiment 2; slurry amended with basalt resulted in statistically higher emissions than the control ($p < 0.05$), while other treatments did not differ statistically.

In experiment 1, total GHG emissions ranged from 1678 to 2146 g CO_2-eq m^{-2} across treatments. Direct nitrous oxide contributed 13–15%, and indirect N_2O contributed 2–3% of total GHG emissions, whereas methane accounted for 83–85%. None of the additive's slurry amended with leonardite, zeolite and bentonite) resulted in statistically lower total GHG emissions than the control slurry.

Similarly, in experiment 2, total GHG emissions ranged from 1833 to 2137 g CO_2-eq m^{-2}. Direct nitrous oxide accounted for 14–17%, and indirect N_2O contributed approximately 5% of total GHG emissions,

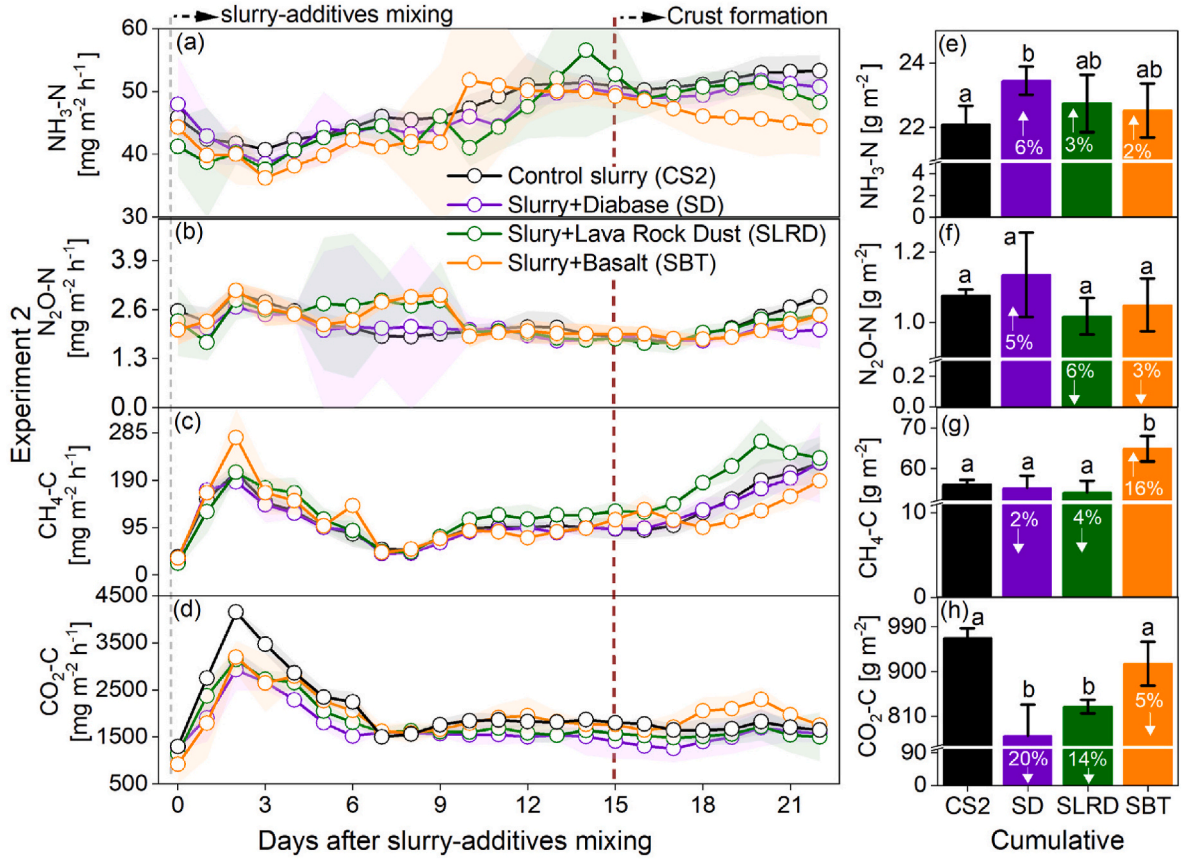


Fig. 5. Daily fluxes (a, b, c, d) and cumulative emissions (e, f, g, h) of ammonia (NH_3-N), nitrous oxide (N_2O-N), methane (CH_4-C), and carbon dioxide (CO_2-C) from experiment 2. Treatments control slurry (CS2) slurry + diabase (SD), slurry + lava rock dust (SLRD) and slurry + basalt (SBT) are shown in black, violet, green and orange colour, respectively. Gray dotted lines mark the day of additive–slurry mixing, and red dotted lines indicate the onset of surface crust formation. Arrows with percentage values on bar graphs indicate the magnitude and direction of change in emissions relative to the control. Mean daily fluxes (g m^{-2} day^{-1}) are shown as coloured symbols with shaded standard deviation (SD) bands, whereas cumulative emissions (g m^{-2}) are shown as bars with whiskers. Shaded bands and whiskers represent ± SD (n = 3). Bars sharing the same lowercase letter are not statistically different ($p > 0.05$), whereas bars with different letters differ statistically ($p < 0.05$) according to Tukey's HSD test. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

Table 2
Cumulative total greenhouse gas (GHG) emissions expressed as CO_2 equivalents (CO_2-eq) and relative contributions (%) of direct nitrous oxide (N_2O), indirect N_2O (estimated from NH_3 volatilisation using the IPCC EF_4 emission factor) and methane (CH_4) for slurry treatments in experiments 1 and 2. Values are means ± standard error (SE; n = 3).

Experiment	Treatment	Direct N_2O contribution (%)	Indirect N_2O contribution (%)	CH_4 contribution (%)	Total GHG ± SE (g CO_2-eq m^{-2})
1	Control slurry (CS1)	14.6 ± 0.4	3.0 ± 0.2	82.8 ± 6.6	1677.9 ± 120.0a
	Slurry + Leonardite (SL)	13.5 ± 0.7	1.6 ± 0.1	85.1 ± 21.0	2146.4 ± 466.3a
	Slurry + Zeolite (SZ)	13.4 ± 1.1	2.0 ± 0.3	84.9 ± 14.9	1849.4 ± 300.1a
	Slurry + Bentonite (SB)	12.5 ± 1.2	2.7 ± 0.3	85.2 ± 3.4	2031.6 ± 96.1a
2	Control slurry (CS2)	16.3 ± 0.2	5.3 ± 0.1	79.3 ± 1.0	1908.0 ± 23.9a
	Slurry + Diabase (SD)	17.2 ± 1.1	5.3 ± 0.2	78.4 ± 2.6	1899.5 ± 71.5a
	Slurry + Lava rock dust (SLRD)	16.0 ± 0.5	5.3 ± 0.2	79.6 ± 2.5	1833.4 ± 57.9a
	Slurry + Basalt (SBT)	14.1 ± 0.6	4.7 ± 0.2	81.9 ± 2.3	2136.8 ± 64.2b

Different letters (a, b) indicate significant differences within each experiment ($p \leq 0.05$).

whereas methane contributed 78–82%. Total GHG emissions from slurry amended with diabase and lava rock dust did not differ statistically from the control slurry. In contrast, slurry amended with basalt resulted in statistically higher total GHG emissions (2137 g CO_2-eq m^{-2}) relative to the control slurry, due to increased CH_4 emissions.

3.6. Elastic net analysis of slurry properties and gaseous emissions

Elastic net regression was applied to the combined dataset, including experiment as a categorical variable. Across gases, ammonia emissions were strongly associated with NH_4^+-N, which had the largest positive coefficient, while OM exhibited a negative association. The model had high explanatory power for NH_3 (adj. $R^2 = 0.98$, Table 3), although this should be interpreted cautiously given the inclusion of experiment as a control factor and the limited sample size. For direct N_2O, the model had moderate explanatory power (adj. $R^2 = 0.46$), with NH_4^+-N, dry matter and pH retained as associated variables. CO_2 emissions were primarily associated with OM (adj. $R^2 = 0.52$, Table 3). Overall, the elastic net analysis indicated stronger relationships between slurry chemistry and NH_3 emissions than for the other gases.

Table 3
Summary of elastic net regression results for log-transformed cumulative ammonia (NH_3), direct nitrous oxide (N_2O), carbon dioxide (CO_2), and methane (CH_4) emissions. The table reports coefficient estimates (Est.), standardised coefficients (β) and model performance (R^2 and adjusted R^2). Predictors not retained in the final elastic net model are indicated by "–". Experiment was included as a categorical adjustment factor during model fitting to account for baseline differences between experimental runs, but its coefficients are not shown because the factor was not interpreted mechanistically. Acidity (pH), total nitrogen (TN), ammonium nitrogen (NH_4^+-N), organic matter (OM), and dry matter (DM) were included as explanatory variables. Asterisks indicate bootstrap selection frequency from stability selection (* ≥ 0.70, ** ≥ 0.80, *** ≥ 0.90). No predictors were retained for CH_4 emissions after penalization.

Predictor	NH_3		Direct N_2O		CO_2		CH_4	
	Estimate	β	Estimate	β	Estimate	β	Estimate	β
(Intercept)	0.64**	–	−0.23*	–	7.02*	–	–	–
pH	–	–	0.05*	0.02	–	–	–	–
NH_4^+-N	2.51***	0.63	0.14**	0.26	–	–	–	–
OM	−0.02***	−0.20	–	–	−0.01***	−0.61	–	–
DM	–	–	−0.03***	−0.20	–	–	–	–
R^2/Adj. R^2	0.99/0.98		0.55/0.46		0.56/0.52		–	

3.7. Cross-validation of NH_3 emission measurement techniques

Cumulative NH_3 emissions derived from the acid-trap method were directly compared with INNOVA measurements for both experiments (Fig. 6a, Fig. S1). In experiment 1, the largest absolute differences between methods were observed for the control slurry and the bentonite treatment, where INNOVA-derived values differed from acid-trap estimates by 1.6 g m^{-2} (14%) and 2.5 g m^{-2} (20%), respectively. In contrast, cumulative NH_3 emissions for slurries amended with leonardite and zeolite were in close agreement between methods, with deviations of −5% and +5% (Fig. 6a). In experiment 2, agreement between the two measurement approaches was consistently high across all treatments. Deviations between acid-trap and INNOVA-derived cumulative NH_3 emissions were 6.6% for the control slurry and within 3% for the diabase-, lava rock dust-, and basalt-amended slurries. Across experiments, cumulative NH_3 emissions estimated by the acid-trap and INNOVA methods did not differ significantly, suggesting good overall consistency.

Across all paired replicates, cumulative NH_3 emissions measured by the acid-trap and INNOVA methods had a strong positive linear relationship (R^2 = 0.95), suggesting high consistency between the two measurement techniques over the observed range of NH_3 emissions (Fig. 6b).

4. Discussion

4.1. Effectiveness of additives for NH_3 mitigation during slurry storage

Across both slurry storage experiments, NH_3 emissions were highest during the early storage phase while subsequent temporal trends differed between the two experiments, with a clearer decline following surface crust development in experiment 1 than in experiment 2. This pattern is consistent with studies showing that physical surface processes strongly influence NH_3 volatilisation during slurry storage. The presence of floating crusts acts as a diffusion barrier, thereby limiting NH_3 transfer to the atmosphere (Prado et al., 2020; Swoboda et al., 2021; Curtis et al., 2023). Thus, amendment effects should be interpreted in the context of these important physical surface processes, particularly crust development.

Under these conditions, amendment-driven NH_3 mitigation was limited. Leonardite reduced cumulative NH_3 emissions by 32%, and among the tested rock powders only zeolite achieved a significant reduction (29%), whereas bentonite, diabase, lava rock dust, and basalt provided no measurable mitigation relative to their respective controls. Ammonia reductions were observed only in experiment 1, whereas no significant treatment effects were observed in experiment 2. Nevertheless, the combined analysis across both experiments retained treatment as a significant source of variation in NH_3 emissions. Similar variability in additive performance has been reported previously for slurry storage systems (Swoboda et al., 2021; Prado et al., 2020; Cao et al., 2022; Khan et al., 2025) and has been attributed to differences in slurry physicochemical properties, amendment characteristics, application rate, and

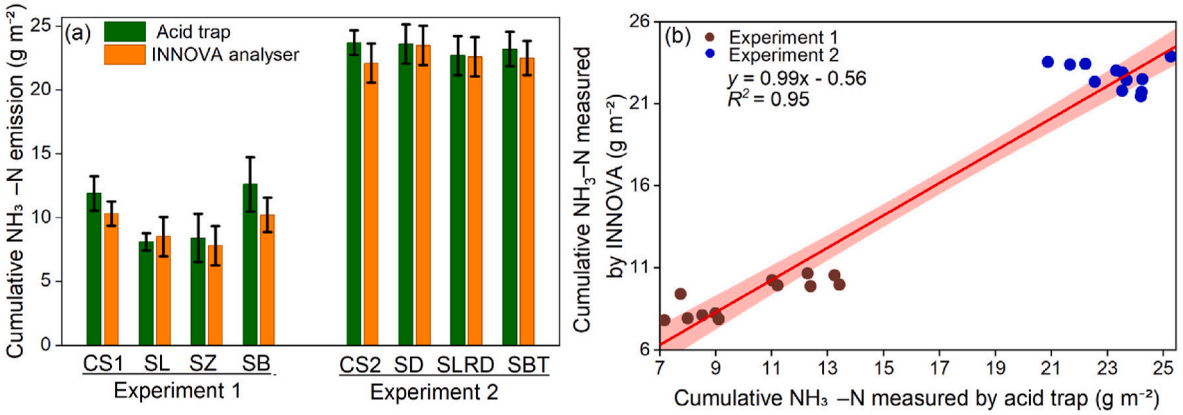


Fig. 6. (a) Comparison of cumulative NH_3–N emissions measured using the acid trap method and the INNOVA photoacoustic gas analyser for each treatment in experiments 1 and 2. Bars represent mean ± SD (n = 3). (b) Linear regression between cumulative NH_3–N emissions measured by the acid trap method and the INNOVA photoacoustic gas analyser using all paired replicates (n = 24). Brown circles represent experiment 1, and blue circles represent experiment 2. The solid red line represents the fitted regression, and the shaded area indicates the 95% confidence interval (R^2 = 0.95). (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

storage conditions that influence NH_3 volatilisation.

The elastic net regression identified NH_4^+-N as the dominant independent predictor of NH_3 emissions, while pH and TN were not retained after accounting for collinearity (Table 3). In view of the established influence of pH on NH_3 volatilisation, the limited importance of pH in the present study may reflect the relatively narrow pH range among control slurry and slurry–additive mixtures (pH 6.4–7.0; Table 1).

Evidence for leonardite-based NH_3 mitigation under slurry storage conditions is limited, as most studies focus on soil incubation systems. For example, Cao et al. (2022) reported NH_3 emission reductions of 32–64%, following 8% leonardite application prior to soil incorporation, a management context not comparable to slurry storage. However, slurry storage studies using other carbon-rich additives provide relevant context. Biochar and lignite-derived materials have been reported to reduce NH_3 emissions during storage by approximately 10–28% (Miranda et al., 2021; Pereira et al., 2022; Viaene et al., 2023; Baral et al., 2023). These findings are consistent with the mitigation observed here for leonardite and support a mechanism where high surface area and reactive functional groups promote NH_4^+ retention within the slurry matrix (Sarıyıldız, 2020; Arrobas et al., 2022; Paul and Datta, 2023; Khalkhal et al., 2025).

Similarly, zeolite achieved a comparable NH_3 reduction (29%) through a mechanistically distinct but well-established pathway. Its high cation-exchange capacity and strong selectivity for NH_4^+ enable direct NH_4^+ sorption and reduce the pool of volatilizable nitrogen (Lefcourt and Meisinger, 2001; Owusu-Twum et al., 2025; Zhuang et al., 2025). Reported NH_3 mitigation by zeolite during slurry storage typically ranges from 10 to 40%, depending on slurry characteristics and application rate (Cole et al., 2007; Lee et al., 2025a), consistent with the magnitude observed here.

In contrast, bentonite, diabase, lava rock dust, and basalt in experiment 2 did not significantly reduce NH_3 emissions compared to control slurry. Although these rock powders are alkaline (De La Villa et al., 2001; Fernández et al., 2009; Gates and Bouazza, 2010; Anh et al., 2017) and caused small increases in slurry pH (Table 1), which could shift the NH_4^+/NH_3 equilibrium toward NH_3 (Prado et al., 2020; Cao et al., 2022; Khan et al., 2025), this did not translate into consistent changes in emissions.

The high explanatory power of the combined model for NH_3 emissions (adj. $R^2 \approx 0.98$) indicates that most observed variability was captured, although partly reflecting systematic differences between experiments. Additional variability may also reflect surface processes and mass-transfer limitations during storage (Wood et al., 2012; Prado et al., 2020; Pedersen et al., 2021; Connolly et al., 2025), as well as competitive cation interactions limiting NH_4^+ retention by minerals with low cation-exchange capacity (Dinuccio et al., 2008; Kupper et al., 2020; Pereira et al., 2022).

While NH_3 reductions have been reported for rock powders such as lava rock dust or basalt, these effects were generally observed at substantially higher application rates (≈8–20% w/w) mainly in solid or poultry manure systems (Witter and Kirchmann, 1989; Kistner-Othmer, 1989; Zaied, 1999). In contrast, studies focusing on untreated slurry storage report weak or inconsistent mitigation for bentonite, diabase, lava rock dust, and basalt, in agreement with the present findings (Swoboda et al., 2021; Curtis et al., 2023).

Overall, NH_3 mitigation during slurry storage was generally limited and differed among additives. Reported mitigation effects in the literature vary substantially with slurry type, application rate, and experimental conditions, and many studies are not directly comparable to storage-relevant slurry systems. These findings highlight the need for systematic, multi-gas evaluations under realistic slurry storage conditions before extrapolating mitigation claims to practice.

4.2. Impact of additives on greenhouse gas emissions (N_2O, CH_4, and CO_2)

4.2.1. General storage dynamics

Immediately after additive incorporation, N_2O emissions remained relatively stable in both experiments (Figs. 4 and 5), suggesting that mixing alone did not stimulate the N_2O-producing processes of nitrification and denitrification during slurry storage (Fangueiro et al., 2013; Prado et al., 2020). When interpreting this response, it should be noted that the slurry had accumulated in the barn collection system for approximately 1–2 weeks and had been mechanically homogenised before sampling. Experimental mixing therefore represented an additional disturbance of previously stored and mixed slurry. This response is consistent with slurry storage conditions that are generally unfavourable for nitrification due to limited oxygen diffusion within the slurry matrix, which restricts nitrate formation and thereby constrains subsequent denitrification processes (Sørensen and Eriksen, 2009; Chadwick et al., 2011; Fangueiro et al., 2013; Prado et al., 2020; Swoboda et al., 2021).

In contrast, CH_4 and CO_2 were associated with strong early responses reflecting storage history rather than additive-specific effects. The sharp CH_4 peak after additive mixing in experiment 2 was likely caused by physical degassing of previously accumulated CH_4 under anaerobic conditions, rather than enhanced methanogenic activity (Sherlock et al., 2002; Dittert et al., 2005). Concurrent increases in CO_2 observed in both experiments likely reflect disturbance-induced release of dissolved CO_2 and transient stimulation of microbial respiration following slurry redistribution (Van Miegroet and Olsson, 2011; Kupper et al., 2020; Prado et al., 2020).

Following surface crust formation, N_2O, CH_4, and CO_2 emissions tended to increase. Previous slurry storage studies suggest that surface crusts may act as diffusion barriers, limiting oxygen penetration from the continuous airflow into the underlying slurry and creating anaerobic or heterogeneous redox microsites that favour methanogenesis and denitrification, thereby influencing CH_4, N_2O, and CO_2 dynamics (Sommer et al., 2000; Hansen et al., 2009; Prado et al., 2020; Swoboda et al., 2021). However, crust characteristics were not quantified in the present study, and therefore the contribution of such mechanisms cannot be confirmed directly.

4.2.2. Direct N_2O emissions

Additive effects on N_2O were generally limited. In experiment 1, leonardite increased cumulative N_2O emissions (+20%) likely due to reduced NH_3 losses and increased mineral N availability for microbial transformation during storage (Brennan et al., 2015; Cao et al., 2022; Khan et al., 2025). This interpretation is supported by the elastic net model, which identified NH_4^+-N as a positive predictor of N_2O emissions (Table 3). In contrast, N_2O emissions under zeolite did not differ from the control despite reduced NH_3 emissions. Previous studies have reported that zeolite can retain NH_4^+ on cation-exchange sites thereby reducing dissolved NH_4^+ availability for nitrification and limiting subsequent NO_3^- formation and N_2O production (Ferretti et al., 2017; Paranaíba et al., 2023; Park et al., 2024a). However, the absence of a corresponding reduction in N_2O emissions in the present slurry-storage experiment indicates that NH_4^+ availability was likely not the sole factor controlling N_2O formation. Under oxygen-limited slurry-storage conditions, the influence of zeolite on N_2O production was likely constrained by redox conditions, localized oxygen availability, and microbial activity rather than by NH_4^+ availability alone (Sørensen and Eriksen, 2009; Chadwick et al., 2011; Fangueiro et al., 2013; Prado et al., 2020; Swoboda et al., 2021).

Bentonite had no consistent effects on N_2O emissions in the present study. Reported mitigation occurred mainly at higher bentonite inclusion rates and in different systems (e.g. ~10% in solid manure or extreme 1:1 ratio in soil; Pratt et al., 2016; Wu et al., 2023). In contrast, the 5% application rate used here did not suppress N_2O formation,

suggesting rate- and matrix-dependence and limited relevance for practical slurry storage conditions.

In experiment 2, N_2O emissions exhibited only weak treatment effects across diabase, lava rock dust, and basalt. To our knowledge, evidence for N_2O mitigation by diabase- or basalt-based materials during raw slurry storage remains limited, with most available studies focused on soil and field applications (Oliveira et al., 2025). In those systems, reported N_2O reductions are generally attributed to mineral weathering–induced pH increases and soil–microbe–plant interactions operating over longer timescales, often at comparatively high application rates (Hinsinger et al., 1995; Blanc-Betes et al., 2021; Chiaravalloti et al., 2023; Oliveira et al., 2025). Such mechanisms are likely less effective during slurry storage (Prado et al., 2020; Swoboda et al., 2021; Khan et al., 2025). Consistent with this, diabase caused no meaningful change in slurry pH relative to the control (Table 1), while the minor pH increases observed for lava rock dust and basalt were likely insufficient to substantially enhance the reduction of N_2O to N_2 during slurry storage, particularly under the heterogeneous and oxygen-limited conditions typical of stored slurry.

4.2.3. CH_4 emissions

Methane emissions reflected both physical release processes immediately after mixing and heterogeneous microbial activity during storage. In experiment 1, cumulative CH_4 emissions increased significantly under bentonite (+25%, Fig. 4c), while leonardite had a non-significant increase associated with high variability among replicates. These responses suggest that early CH_4 dynamics were influenced more by mixing-induced gas release than by measured bulk slurry properties. Mineral additives such as zeolite and bentonite may alter slurry structure and gas transfer through sorption and exchange processes under anaerobic storage conditions (Swoboda et al., 2021). Leonardite, in contrast, is a carbon-rich amendment that may provide additional fermentable substrates under anaerobic conditions (Prado et al., 2020; Cao et al., 2022; Khan et al., 2025). Consistent with these observations, the combined elastic net model retained no significant predictors for CH_4 emissions (Table 3), suggesting that methane variability was not explained by the measured bulk slurry properties.

In experiment 2, a pronounced CH_4 peak immediately after mixing was observed. Following this initial peak, treatment effects on cumulative CH_4 emissions were modest but detectable, with basalt resulting in a significant increase (+16%) relative to control. Previous studies suggest that enhanced microbial activity may explain higher CH_4 emissions from rock powder-amended cattle slurry, as nutrient-limited microbial growth can be stimulated by mineral nutrient release (Carson et al., 2007; Li et al., 2020; Swoboda et al., 2021). In addition, mineral surfaces can promote selective microbial colonization, consistent with the "mineralosphere" concept, and thereby influence microbial activity (Carson et al., 2009; Uroz et al., 2015). Basalt also undergoes microbially mediated weathering and releases mineral nutrients that may further stimulate microbial processes (Silva et al., 2013). However, because microbial communities, dissolved mineral nutrients, and redox conditions were not measured in the present study, these mechanisms remain speculative and could not be confirmed directly.

Furthermore, no predictors were retained by the elastic net model for CH_4 emissions (Table 3), suggesting that methane dynamics were driven primarily by transient physical processes and heterogeneous microbial activity rather than by measured bulk slurry properties.

The weak relationship between CH_4 emissions and measured bulk slurry properties further suggests that methane dynamics were not governed by average slurry chemistry alone. Methane production and oxidation can occur simultaneously within spatially distinct anaerobic and aerobic microsites, where localized redox (Eh) conditions regulate methanogenic activity (Duan et al., 2017; Beegum et al., 2026). In addition, local availability of fermentable carbon from carbon-rich amendments such as leonardite may stimulate methanogenesis in specific regions of the slurry, while temporal lags between substrate availability and microbial responses may further weaken relationships between bulk physicochemical properties and cumulative CH_4 emissions (Prado et al., 2020; Cao et al., 2022; Khan et al., 2025). These interacting processes may explain why no significant physicochemical predictors of CH_4 emissions were identified by the elastic net model.

4.2.4. CO_2 emissions

Carbon dioxide emissions exhibited weaker and less consistent responses to additives than CH_4, suggesting partial decoupling between carbon mineralisation and methanogenesis during slurry storage (Swoboda et al., 2021; Thorn et al., 2022; El Bied et al., 2023). In experiment 1, no significant differences in cumulative CO_2 emissions were observed among treatments, despite treatment-related variation in CH_4 emissions. This suggests that additive effects were more strongly reflected in methanogenic processes than in overall CO_2 release during storage.

In experiment 2, diabase and lava rock dust significantly reduced cumulative CO_2 emissions. As pH and N content were similar for all additives of experiment 2, and as the elastic net model did not retain nitrogen-related variables for CO_2, these experiment-specific reductions cannot be attributed to consistent effects of nitrogen availability. Thus, one plausible explanation is enhanced retention of CO_2 in the aqueous phase or increased carbonate buffering associated with mineral-derived constituents, as reported in mineral–water systems and slurry studies (Matter et al., 2007; Hodges et al., 2021; Swoboda et al., 2021).

Overall, additive effects on N_2O, CH_4, and CO_2 emissions during slurry storage were inconsistent among gases and treatments. Emission dynamics were influenced not only by additive properties, but also by storage-phase processes such as mixing, gas release, and crust development. These findings highlight the importance of evaluating slurry additives within an integrated multi-gas framework under realistic storage conditions.

4.3. Trade-offs and implications for total greenhouse gas mitigation

A central concern in NH_3 mitigation during slurry storage is nitrogen trade-offs, where conserved N is redirected into other gaseous pathways (Stevens and Quinton, 2009; Khan et al., 2025). In the present study, this pattern was observed for leonardite, which reduced NH_3 emissions while coinciding with significantly higher N_2O release. This observation suggests that retained N remained bioavailable and was subject to microbial transformation, particularly in later storage stages following crust formation. However, because nitrogen transformation processes were not measured directly, this link remains associative rather than causative. Similar co-occurrence of NH_3 mitigation and increased N_2O emissions has been reported in other slurry storage studies, where retained mineral N was redistributed within the emission cascade rather than removed entirely (Prado et al., 2020; Cao et al., 2022; Khan et al., 2025).

In contrast, zeolite reduced NH_3 emissions without a corresponding increase in N_2O, suggesting no detectable trade-offs. This divergence suggests that zeolite-based mitigation may involve stronger physicochemical retention of NH_4^+, such as cation-exchange processes that reduce microbial accessibility and limit downstream nitrogen transformations (Ferretti et al., 2017; Paranaíba et al., 2023; Park et al., 2024b), although direct confirmation of these mechanisms was beyond the scope of this study.

In experiment 2, where no amendment effectively reduced NH_3 emissions, nitrogen retention within the slurry matrix was limited and N_2O responses remained weak. This supports that trade-offs are not an inherent consequence of additive application but arise when NH_3 mitigation sustains bioavailable N under storage conditions (Khan et al., 2025).

From a climate perspective, total greenhouse gas emissions were dominated by CH_4, with N_2O contributing a smaller but more potent share, while CO_2 was considered biogenic and therefore excluded from

climate accounting (Liu and Greaver, 2009; Prado et al., 2020; Cao et al., 2022). Estimated indirect N_2O emissions associated with NH_3 volatilisation accounted for an additional approximately 2–5% of total greenhouse gas emissions. Thus, small CH_4 increases can offset NH_3 mitigation benefits. Consistent with this, additives that reduced NH_3 emissions, such as leonardite and zeolite, did not result in net reductions in CO_2-equivalent emissions because CH_4 emissions increased or remained unchanged (Table 2). Although reduced NH_3 emissions under leonardite and zeolite also decreased the estimated indirect N_2O contribution, this additional climate benefit was relatively small and did not alter the overall greenhouse gas balance. Conversely, treatments that failed to mitigate NH_3 also did not reduce total greenhouse gas emissions, underscoring that effective mitigation during slurry storage requires concurrent control of both nitrogen and carbon pathways rather than targeting NH_3 alone.

Beyond direct emissions during storage, NH_3 volatilisation also represents a loss of plant-available N from the agricultural nutrient cycle that is typically compensated through mineral fertilizer production via the Haber–Bosch process. Industrial production of mineral N fertilizers is energy-intensive and associated with substantial greenhouse gas emissions. For example, approximately 3.6 kg CO_2-equivalent are emitted during the production of 1 kg mineral N in ammonium fertilizers (Yara International ASA, 2023). Thus, NH_3–N losses may translate into several kg CO_2-equivalent if replaced by synthetic fertilizer, although this is approximate.

Overall, these findings highlight that NH_3 mitigation strategies for slurry storage must be evaluated within an integrated, multi-gas framework. Mitigation that does not constrain microbial processes may redistribute emissions rather than reduce them, reinforcing the need to account for pollution swapping and greenhouse gas trade-offs under storage conditions.

4.4. Methodological considerations for NH_3 measurement

Although direct comparisons between acid traps and optical NH_3 analysers under slurry storage conditions are limited, the observed differences reflect their measurement principles. Acid traps integrated NH_3 emissions over the sampling period, while the INNOVA photoacoustic analyser measured NH_3 concentrations at discrete time points. Consequently, the observed differences between the two methods may partly reflect differences in temporal sampling rather than inherent differences in measurement accuracy, particularly during periods of rapidly changing NH_3 emissions following slurry mixing (Ndegwa et al., 2009; Khan et al., 2025).

In the present study, no statistically significant differences were detected between the optical analyser and acid trapping, although numerical deviations were observed, reaching up to ~20% in experiment 1 and ~7% in experiment 2 (Fig. 6a). These relatively small differences indicate that, under controlled slurry storage conditions with comparatively stable emission profiles, optical NH_3 measurements can yield estimates comparable to time-integrated trapping methods. However, the absence of statistically significant differences should not be interpreted as full methodological equivalence. Acid traps inherently smooth short-term variability, whereas optical measurements remain sensitive to instrument equilibration, sampling frequency, and transient concentration changes, particularly immediately after slurry disturbance.

Overall, this study provides an initial validation of optical NH_3 measurement under slurry storage conditions. Despite good agreement, further evaluation across a broader range of slurry types, storage systems, and field conditions is required before optical analysers can be considered a general substitute for acid trapping methods.

4.5. Limitations and applicability under practical slurry storage conditions

The present study was conducted under controlled laboratory conditions using gas-tight dynamic flow-through chambers operated under near-ambient pressure. Fresh air was continuously supplied at a flow rate of 2 L min^{-1} over the slurry surface, and outlet air was continuously discharged through the measurement system, providing standardized and reproducible conditions for quantifying gaseous emissions. Nevertheless, unlike practical slurry storage systems, the experimental setup operated under constant temperature and controlled airflow and was not exposed to environmental factors such as wind, precipitation, solar radiation, or seasonal temperature fluctuations. These factors can influence gas transfer, surface crust development, microbial activity, and consequently the magnitude of gaseous emissions and the apparent effectiveness of mitigation additives. Therefore, the mitigation efficiencies observed in this study should be interpreted as comparative results obtained under standardized laboratory conditions rather than as direct predictions of field-scale performance. Further validation under commercial slurry storage conditions is required to assess the robustness and practical applicability of these findings.

Beyond these environmental influences, additive performance at farm scale will also depend on how additives are incorporated during storage. In this experiment, additives were mixed once with a fixed slurry batch at 5% (w/w), with no subsequent slurry additions. In practice, continued filling with untreated slurry would progressively dilute a single initial dose and could alter its mitigation effectiveness. Potential application strategies include dosing incoming slurry during transfer to storage or making repeated additions with sufficient mixing to distribute the additive. However, the suitability of these strategies, the persistence of additive effects, and the consequences of repeated mixing for gaseous emissions require evaluation under practical farm conditions.

In addition, the mechanistic explanations presented in this study are based on the observed emission patterns and supported by findings from the existing literature, as direct measurements of microbial communities, functional genes, enzyme activities, or temporal changes in ammonium and nitrate concentrations were beyond the scope of this study. Because NH_4^+ and NO_3^- concentrations were not measured repeatedly during storage, temporal nitrogen transformations could not be assessed indirectly. Future studies should combine gaseous emission measurements with microbial analyses, such as 16S rRNA sequencing, quantification of functional genes involved in nitrification, denitrification, and methanogenesis, together with repeated measurements of ammonium and nitrate concentrations during slurry storage, to better elucidate the mechanisms underlying additive-induced changes in gaseous emissions.

In addition, the present study evaluated additive performance during a 22-day controlled storage period, which was designed to capture short-term responses following additive incorporation, including early emission dynamics and initial crust development. This is relevant because these additives are intended to be mixed with slurry before field application (Cao et al., 2022; Khan et al., 2025). Evaluating their performance during the storage phase therefore provides important information on their ability to reduce emissions before slurry is applied to land, where NH_3 emissions typically peak immediately after application and decline rapidly within the first few days (Ni et al., 2015; Zhang et al., 2018; Khan et al., 2025). Nevertheless, longer storage periods may further influence slurry physicochemical properties, microbial activity, crust development, and cumulative gaseous emissions. Future studies should therefore investigate the long-term persistence of additive effects under extended commercial slurry-storage conditions.

5. Conclusion

This study demonstrates that the effectiveness of mineral and humic additives for emission mitigation during slurry storage was generally limited and differed among additives and gaseous products. Moderate NH_3 reductions were observed for leonardite and zeolite, whereas the

other rock-based additives provided little or no mitigation. Where NH_3 mitigation occurred, retained nitrogen was not necessarily environmentally benign, as illustrated by increased N_2O emissions under leonardite, highlighting potential nitrogen trade-offs during storage. In contrast, zeolite reduced NH_3 emissions without increasing N_2O emissions, suggesting more favourable mitigation performance.

The combined elastic net analysis indicated that NH_3 emissions were most strongly associated with NH_4^+-N availability, whereas CH_4 and CO_2 emissions exhibited limited or inconsistent relationships with the measured bulk slurry properties. Across all gases, emission dynamics were also influenced by physical storage processes such as mixing and crust formation that were not fully represented by the measured bulk slurry properties.

In addition, the close agreement between optical NH_3 measurements and conventional acid trapping under controlled storage conditions supports the use of high-frequency optical methods for slurry studies, while highlighting the need for further validation across a broader range of storage systems and field conditions. Overall, these findings emphasise the importance of evaluating slurry amendments within a multi-gas framework that considers NH_3 mitigation together with potential nitrogen trade-offs and greenhouse gas responses. Further research across different slurry types, storage durations, and amendment application rates is required to identify when mineral and humic additives may provide consistent environmental benefits.

CRediT authorship contribution statement

Fawad Khan: Writing – original draft, Methodology, Formal analysis, Conceptualization. **Waqas Qasim:** Writing – review & editing, Methodology, Formal analysis, Conceptualization. **Benjamin Wolf:** Writing – review & editing, Validation, Formal analysis. **Deise Aline Knob:** Project administration, Methodology. **Lutz Breuer:** Writing – review & editing, Project administration. **Klaus Eder:** Writing – review & editing, Resources. **Lindsay Dawn Thurman:** Data curation. **Andreas Gattinger:** Writing – review & editing, Visualization, Supervision, Resources, Project administration, Conceptualization.

Declaration of competing interest

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

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.jclepro.2026.149870.

Data availability

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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