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Effect of slurry bio-acidification and leonardite addition on ammonia and greenhouse gas emissions in soil-plant mesocosms

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ABSTRACT

Greenhouse gas and ammonia emissions from organic and mineral fertilizer application in agriculture contribute significantly to ecosystem eutrophication and global warming. To reduce ammonia emissions from slurry, acidification using sulfuric acid is one of the most effective practices, but the costs for machinery as well as requirements concerning environmental and labour safety are a challenge for small farms. For this reason, there is a rising interest in sustainable and cost-effective alternative slurry amendments, particularly bio-acidification and mineral amendments. Research on the subject has focused primarily on reducing ammonia and greenhouse gas emissions during slurry storage, leaving the impact on emissions following fertilizer application to soil-plant systems largely unexplored. Therefore, this study evaluates the effect of the three different slurry amendments cheese whey, sauerkraut juice, and leonardite on ammonia, nitric oxide, and greenhouse gas (methane, nitrous oxide, carbon dioxide) emissions from soil-plant mesocosms. After slurry application, emissions were measured for nine days using an automated incubation system and mesocosms received 80 kg N ha^{-1} untreated slurry or a mixture of slurry and amendment. Amending slurry with cheese whey significantly reduced ammonia emissions by 91 % and nitrous oxide by 23 %. Sauerkraut juice significantly decreased ammonia emissions by 92 % but significantly increased nitrous oxide emissions compared to slurry treatment. Leonardite reduced ammonia and nitrous oxide emissions insignificantly. These findings suggest that bio-acidification of slurry using cheese whey or sauerkraut juice is the most effective method for reducing ammonia emissions following fertilizer application in soil-plant mesocosms. A large variability particularly of nitrous oxide, nitric oxide and ammonia emissions from soil cores receiving unamended fertilizer indicated the importance of field-scale experiments to further verify the findings at larger scales and at natural soil environmental conditions.

1. Introduction

At present, the growing world population drives a growing demand for agricultural products, including milk and meat. Livestock farming produces manure containing nitrogen (N) which is used as fertilizer in crop production and has potential to substitute a significant share of energy-intensive synthetic N fertiliser production (Luo et al., 2022; Ren et al., 2023; Taube et al., 2024). In pig and cattle slurry, approximately 50 % of the total N content is dissolved ammonium (NH_4^+) which is in chemical equilibrium with gaseous ammonia (NH_3; Sommer et al., 1993). It is well established that NH_3 volatilization during manure storage and surface application of fertilizers can be substantial (Bouwman et al., 2002; Ntinas et al., 2013), and that approximately 90 % of global NH_3 emissions originate from agricultural sources (Anderson et al., 2003; Covali et al., 2021). These emissions contribute significantly to the formation of particulate matter ($PM_{2.5}$) in the atmosphere through chemical reactions with acidic compounds such as sulfur dioxide (SO_2) and nitrogen oxides (NO_x), posing severe air quality and public health risks (Bittman et al., 2014). Furthermore, the deposition of atmospheric NH_3 emissions and its reaction products indirectly affects greenhouse gas (GHG) and N trace gas emissions by introducing additional N into soils. This added N stimulates soil N turnover processes producing

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nitrous oxide (N_2O) and nitric oxide (NO; Butterbach-Bahl et al., 2013; Kavanagh et al., 2021; Gioelli et al., 2022). In addition, storage and application of manure releases methane (CH_4) and N_2O to the atmosphere, with manure management contributing up to 9 % of global GHG emissions and in turn contributing to global warming (Gerber et al., 2013; Dennehy et al., 2017). Besides the global warming effect, N pollution through NH_3 emissions poses significant risks to human health and leads to eutrophication as well as biodiversity loss (Buoio et al., 2023; He et al., 2023). Thus, effective mitigation strategies are crucial to reduce NH_3 and GHG emissions from slurry, which will also help to optimize slurry nutrient content, sustain agricultural yields, and preserve ecosystem integrity (Prado et al., 2020; Kavanagh et al., 2021; Regueiro et al., 2022).

One frequently used method to reduce NH_3 emissions is slurry acidification (Fangueiro et al., 2015; Geels et al., 2023). Slurry acidification involves lowering the pH of livestock manure by adding acids such as sulfuric acid (H_2SO_4), shifting the chemical equilibrium between ammonium (NH_4^+) and NH_3 (Fangueiro et al., 2015). This process favours ammonium retention in the slurry, reducing NH_3 volatilization into the atmosphere (Prado et al., 2020; Keskinen et al., 2022). Studies have shown that slurry acidification can cut NH_3 and methane emissions by up to 75 % and 61 %, respectively (Misselbrook et al., 2016; Overmeyer et al., 2021). Supporting this, a meta-analysis suggested that acidification reduced NH_3 emissions by 69 % during storage and application, and additionally reduced emissions of N_2O by 21 %, CH_4 by 86 %, and CO_2 by 15 % without negative effects of pollution swapping (Emmerling et al., 2020). The most frequently used agent for acidification is sulfuric acid (H_2SO_4). However, this substance is problematic since it requires training in dealing with hazardous substances and expensive machinery for the application, as H_2SO_4 is corrosive, and it generates foam when mixed with slurry. Other undesirable effects include potential for soil acidification followed by displacement of nutrients from cation exchange sites, inhibition of anaerobically active microorganisms, formation of volatile sulfur-containing compounds, and its ban in organic farming (Borst, 2001; Hjorth et al., 2015; Prado et al., 2020). Consequently, there is a need for alternatives to H_2SO_4 (Fangueiro et al., 2013; Loide et al., 2020; Prado et al., 2020; Gioelli et al., 2022; Regueiro et al., 2022).

Bio-acidification involves producing organic acids in slurry by adding microbes, by adding readily degradable organic substrates that are converted into organic acids by the existing microbial community in the slurry, or by using acidic sub-products from fermentation processes in the food industry (Clemens et al., 2002; Nykänen et al., 2010; Prado et al., 2020; Regueiro et al., 2022). Such acidic substances include cheese whey and sauerkraut juice, which are abundant in industrialized countries such as Germany, where milk processing companies and canned food manufacturers produce 4 million tons of cheese whey and 1300 m^3 of sauerkraut juice annually (Germany Trade and Invest, 2018; Statista Research Department, 2023). These organic acids have no economic value and are available free of charge upon request. Valorising these products can be cost-effective in the long run, similar to technologies such as anaerobic digesters for biogas production, fermentation units for lactic acid extraction, and advanced filtration systems for bioactive compound separation (Pereira et al., 2002; Pires et al., 2021). However, finding ecologically appropriate solutions to treat and dispose of these "waste" substances often involves significant upfront costs and resource-intensive processes (Saba et al., 2023; Estikomah and Masykuri, 2023).

Several studies on bio-acidification of slurry have shown positive results, like Regueiro et al. (2022), who demonstrated that bio-acidification using fermentable substrates (glucose, brown juice extracted from biomass, or a combination) reduced CH_4 emission during pig slurry storage. The study of Gioelli et al. (2022) demonstrated that adding whey to slurry significantly lowered CH_4 emissions by 54 % during slurry storage. Similarly, Prado et al. (2020) showed that adding cheese whey and sugar to dairy farm slurry reduces NH_3 volatilization by 68 % and 45 %, respectively, and Sepperer et al. (2021) found that the addition of cheese whey and milk to cattle slurry keeps its pH below 5, thereby reducing NH_3 loss.

Thus, using by-products of other production processes for bio-acidification of manure or slurry can create synergies between the effective management of organic acid waste and the protection of climate and environment through mitigating NH_3 and GHG emissions in agricultural productions systems (Prado et al., 2020; Gioelli et al., 2022; Athar et al., 2023; Garder et al., 2023).

Apart from bio-acidification, slurry treatment using amendments like stone powder or organic substances with a high carbon content and wide carbon-to-nitrogen ratio such as wheat straw, sawdust, or leonardite, which promote microbial immobilization and help to reduce NH_3 emissions (Li et al., 2013; Chang et al., 2019). These amendments provide additional carbon sources that enhance microbial growth and offer a larger surface area for ammonium adsorption, further promoting microbial immobilization thereby reducing N losses through volatilization (Wei et al., 2020; Cao et al., 2022). Specifically, leonardite decreased NH_3 loss by 32–64 % and N_2O emission by 33–77 % compared to a pure slurry application (Li et al., 2021; Cao et al., 2022). Furthermore, incorporating such organic amendments can enhance soil structure, increase water retention, and promote long-term soil fertility, making them a sustainable option for integrated manure management systems (Arrobas et al., 2022; Cao et al., 2022; Piri et al., 2023).

Most mentioned studies on the emission reduction of NH_3 and GHG associated with slurry have primarily focused on slurry storage (Fangueiro et al., 2015; Loide et al., 2020; Prado et al., 2020; Gioelli et al., 2022; Regueiro et al., 2022), with so far limited attention given to emission reductions following fertilizer application. This gap limits identification of relevant N loss pathways induced by NH_3 reduction. One such phenomenon, "pollution swapping", is characterized by increased N_2O emissions due to higher mineral N availability attributed to reduced NH_3 loss from applied slurry. Though identifiable through field experiments, such measurements of NH_3 and GHG emissions are labour and cost intensive. This motivates alternative laboratory design experiments prior to field campaigns. On a laboratory scale, soil-plant mesocosms can be used to assess the influence of slurry amendments on gas fluxes after the application of treated slurry in order to screen several amendments prior to extensive field experiments. However, manual gas sampling for determination of GHG emissions is common in the lab and results in low temporal resolution. Automated incubation systems allow higher measurement frequency, but plant-free soil samples are often used due to insufficient lighting in incubation cabinets.

One well-established method for NH_3 measurement in laboratory experiments is NH_3 capture in acid traps. However, the time resolution of the measurements depends on the frequency at which traps are changed manually, and subsequent laboratory analysis of the trap liquid is time-consuming. The advent of optical spectroscopy for trace gas analysis offers simultaneous determination of the mixing ratios of several trace gases at a temporal resolution of 0.25–1 Hz, potentially making subsequent analysis of acid trap liquid unnecessary. However, such instruments are expensive and require a custom-made incubation system designed for the automation of measurements. Until now, comparisons between different approaches for measuring NH_3 emissions in laboratory-scale experiments have been scarce.

Therefore, this study aims to: (i) assess the impact of cheese whey, sauerkraut juice, and leonardite on ammonia emissions of soil-plant mesocosms; (ii) monitor the response of other trace gases to treated slurry addition to identify potential pollution swapping; (iii) measure the impact on greenhouse gases and global warming potential by use of an efficient automatic soil-plant incubation system with a lighting system for each mesocosm; and (iv) compare ammonia fluxes using different measurement approaches.

2. Materials and methods

2.1. Soil mesocosms

The soil mesocosms used in this study were obtained from an agricultural field located in Selters, Germany, approximately 6 km from the Research Farm Gladbacherhof, Justus-Liebig University Giessen. The site is in the temperate climatic zone, with an average annual temperature of 9.3 °C and a mean annual precipitation of 655 mm. The soil was classified as a Haplic Luvisol (Schulz et al., 2014). The soil properties of the field were determined from four soil profiles (50 cm wide, 60 cm deep). Samples for determination of SOC, TN and pH were collected from 0 to 10, 10–30, and 30–60 cm layers by collecting a bulk sample across the layer face to ensure representativity. For the layer of 0–10 cm, average pH was 5.43, bulk density was 1.45 g cm^{-3}, average soil organic carbon content (SOC) 1.04 %, and total nitrogen (TN) was 0.13 %. The full dataset is presented in supplementary material (Table S1). For this study, a total of 54 soil cores were collected across three experiment, with 18 cores per experiment taken from six distinct sampling spots, and three soil mesocosms taken in close proximity at each spot. For each experiment, fresh soil cores were collected from the site. To collect the soil mesocosms, polymethylmethacrylate cylinders (127 mm diameter, 260 mm height; SAHLBERG GmbH & Co. KG, Germany) were inserted 0.16 m into the soil. A lid was tightly fixed at the bottom to prevent soil loss during transport and leaching during experiments. In the laboratory, small plants and roots were removed with minimal disturbance to prepare the soil for ryegrass seed germination. The ryegrass seeds were soaked in water for 24 h, then placed on moistened paper towels in trays covered with parafilm. After one week, three seedlings were transplanted per core at a depth of 2–3 cm, which is equal to a common sowing rate of 250 germinable grains m^{-2}. Plants were grown for 15 days before the start of the experiment under controlled laboratory (25 °C) conditions.

2.2. Slurry and additives

The cattle slurry used in this study originated from an organically managed farm in southern Germany. The sauerkraut juice was obtained from a canned food producer in southern Germany, and the cheese whey was collected from a cheese dairy also located in southern Germany. Technical problems in the soil-plant automatic incubation systems delayed the start of experiments 2 and 3, so that fresh slurry was obtained for these experiments, with the slurry compositions being provided (Table 1). All samples (slurry, sauerkraut juice, and sour cheese whey) were stored in plastic barrels at 4 °C until start of the experiment. Leonardite was purchased from a construction and mining company located in southern Germany, and stored in a controlled laboratory environment at 25 °C.

2.3. Initial titration experiment for adjustment of pH and nitrogen

An effective reduction of NH_3 emissions from slurry was previously reported for pH 5.5 (Fangueiro et al., 2015). To determine the amount of amendments required to adjust the slurry pH to these values, titration experiments were conducted. A pH of 5.5 could only be achieved by mixing slurry with at least the same volume of amendment. To make the amendments more practical, a pH level of 6.5 was targeted and achieved using a 2:1 slurry to cheese whey, and slurry to sauerkraut juice ratio. Based on the N content, the application rates for both the slurry and slurry-amendment mixture were calculated to achieve the application rate of 80 kg N ha^{-1} (Table 1). For experiment 1, slurry and cheese whey were thoroughly mixed in their barrels and sufficient quantities of liquid manure or amendment to apply the slurry and the slurry-amendment mixture to all mesocosms were taken from their respective barrels, mixed and applied. The same procedure was followed when using sauerkraut juice as slurry amendment (experiment 2). For experiment 3, on the morning of application, the slurry was thoroughly mixed in its barrel; the sufficient quantities of slurry for all mesocosms were transferred into a vessel, and the manufacturer-recommended amount of leonardite (2 % mass) was added and mixed into the slurry.

Table 1
Chemical characteristics and application rate of slurry (S), amendments cheese whey (W), sauerkraut juice (SJ), and leonardite (L) and their mixtures for experiments 1, 2, and 3, respectively. Please note that technical problems in the soil-plant incubation systems delayed the start of experiments 2 and 3, thus fresh slurry was obtained for the named experiments.

Experiment	Type	pH	Total N (kg N t^{-1})	NH_4^+ (kg N t^{-1})	Organic content (kg t^{-1})	Application rate [ml]
1	Slurry (S)	7.0	2.5	1.1	46.30	40.5
	Cheese whey (W)	3.4	1.3	0.35	28.00	
	Slurry + Cheese whey (SW)	6.5	2.1	0.24	13.60	48.3
2	Slurry (S)	7.5	2.0	1.4	14.20	50.68
	Sauerkraut juice (SJ)	3.7	1.4	0.82	24.70	
	Slurry + Sauerkraut juice (SSJ)	6.5	1.7	1.1	28.40	59.62
3	Slurry (S)	7.5	2.0	1.4	14.20	50.68
	Slurry + Leonardite (SL)	7.0	2.7	1.6	39.20	37.54

2.4. Soil-plant mesocosm incubation

In this study, an automated soil-plant mesocosm incubation system was used for continuous monitoring of soil-atmosphere trace gas exchange under controlled laboratory conditions. The system comprises two thermostatic cabinets which maintained the temperature at 18 °C during the experiments (Lovibond ET 651-8, Tintometer GmbH, Dortmund, Germany). Each cabinet accommodated 9 soil-plant mesocosms with a headspace height of 0.05 m. Particularly NH_3 and N_2O emissions exhibit significant spatial and temporal variability; therefore, the number of replicates should be maximized. At the same time, space in incubation cabinets is limited, so that an experimental design with two control groups (unfertilized and pure slurry) and three groups with treated fertilizer only allows three replicates. For this reason, three independent experiments were conducted with each experiment using 18 soil-plant mesocosms. In each experiment, six mesocosms received pure slurry (S), six received slurry amended with specific materials, and six served as the unfertilized control (C). In the first experiment, the slurry amendment was cheese whey (SW), in the second experiment, it was sauerkraut juice (SSJ), and in the third experiment, it was leonardite (SL, Fig. 1). Details of the specific slurry and slurry-amendment mixtures used in each experiment are provided (Table 1). As soils need to be accessible with heavy machinery when spreading slurry, the water-filled pore space (WFPS) was set to 50 % across all experiments, similar to the WFPS found in the field during fertilizer application. After adjusting the WFPS of soil-plant mesocosms, they were transferred into the incubation cabinets, which were maintained at 65 % relative humidity with a 12-h day-night cycle. Day-night cycles were generated using LED lights (LEDVANCE, Parkring 33, 85748 Garching, Germany). After a three-day pre-incubation period during which soil-plant mesocosms were maintained at 50 % WFPS, slurry, as well as slurry-amendment mixtures (SW, SSJ, SL), were applied, with the application technique mimicking a drag-hose technique. Since several studies showed that NH_3, and GHG emissions peak and drop rapidly within the first few days after fertilizer application (Ni et al., 2015; Zhang et al., 2018), mesocosms were monitored for 9 days in the incubation system.

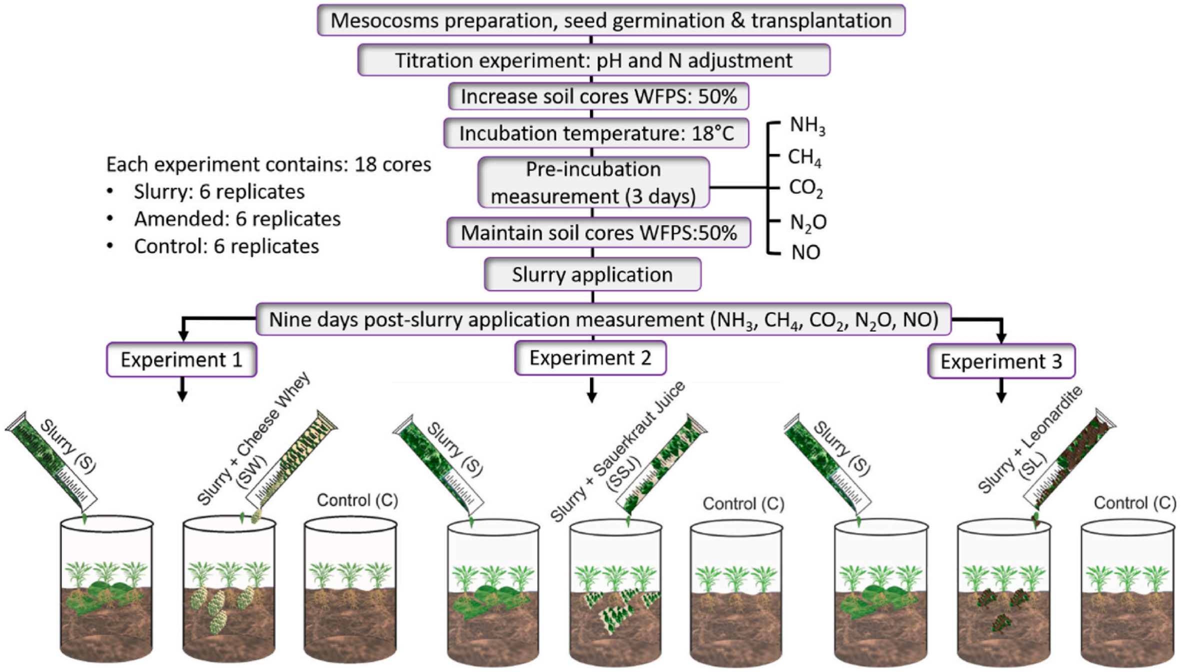

Fig. 1. Overview of the design of the three distinct and separate experiments. In each experiment, three treatments were compared, with six soil cores assigned to each treatment: the pure slurry treatment (S), the control treatment (C) and the slurry plus amendment treatment. The slurry plus amendment treatments in experiments 1, 2 and 3 were slurry with cheese whey (SW), slurry with sauerkraut juice (SSJ), and slurry with leonardite (SL).

2.5. *Measurement of trace gas fluxes*

All trace gas fluxes were measured using the dynamic chamber approach (Brümmer et al., 2008; Pape et al., 2009; Subramaniam et al., 2024) and NH_3 flux was additionally measured using acid traps. In the dynamic chamber approach, the headspace of an incubation chamber containing a soil-plant mesocosm is flushed with a gas of which the trace gas concentration is measured. Since production of the trace gas in the

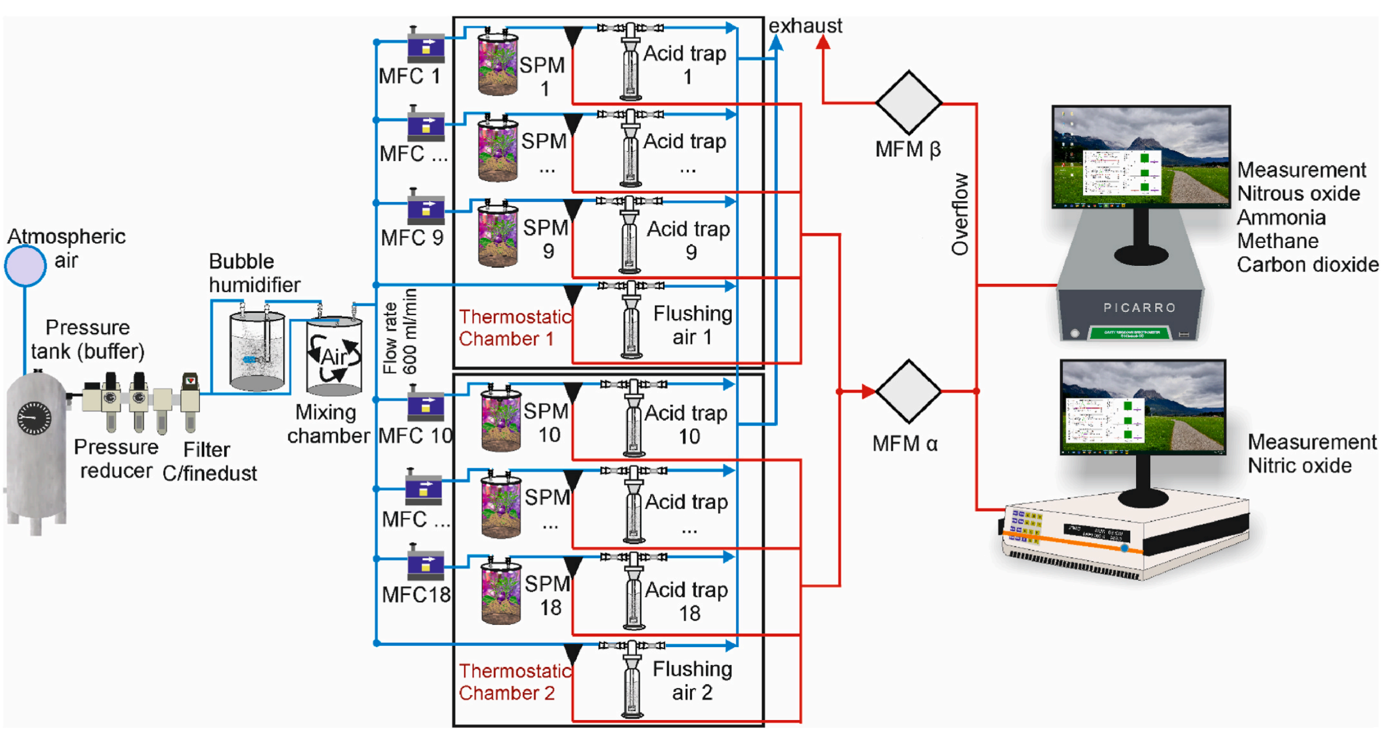

Fig. 2. Schematic diagram of automated soil incubation system. Blue lines represent gas flow pathways (6 mm polytetrafluoroethylene tubes) for default position of 3/2-way valves (black triangles). Mass flow controllers (MFC) regulate the continuous air stream for each soil-plant mesocosm (SPM) to 600 ml min^{-1}. Red lines show gas flow when valve is switched, directing gas of an individual SPM to the trace gas analysers. The CRDS and CLD analysers determine NH_3, CH_4, CO_2, N_2O, and NO, respectively. Acid traps for NH_3 flux determination consist of a washing bottle filled with 100 ml of 0.1M oxalic acid. Mass flow meters (MFM) α and β are for verification. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

soil-plant-mesocosm increases the trace gas mixing ratio of the flushing gas, the trace gas flux can be calculated from the difference in trace gas mixing ratio of the flushing gas before and after passing the chamber headspace. In this study, pressurized ambient air from a storage tank was used as the flushing gas, with humidity level regulated to 65 % using Milli-Q water (RH/T probe HC2-S3C03, ROTRONIC Messgeräte GmbH, Germany, Fig. 2) and the continuous flow through each soil-plant mesocosm being regulated to 600 mL min^{-1} using mass flow controllers (MFCs; Bronkhorst High-Tech B.V; Fig. 2). In its default position, the valve downstream of each soil-plant mesocosm (Fig. 2) directed the air flow through the acid traps consisting of an individual gas-washing bottle made of borosilicate glass (ROBU VitraPOR Borosilicate Glass 3.3, Hattert, Germany). These gas-washing bottles, with a height of 0.1 m and a capacity of 100 mL, were filled with a 0.1 M oxalic acid solution capturing NH_3 in the form of dissolved NH_4^+. Acid traps were sampled daily for 4 days after fertilizer application, and every second day thereafter as recommended by Zuazo (2016). Ammonium concentration was determined by indophenol colorimetry using an Epoch Microplate Spectrophotometer (BioTek Instruments Inc., United States; Bolleter et al., 1961).

A customized computer program controlling the 3/2-way-valves (Fig. 2) sequentially switched the valves to their alternative position, directing the gas flow of a single soil-plant mesocosm to the analytical instruments. Since NH_3 is an adhesive molecule, a certain period of time must be allowed during which the NH_3 concentration of the mesocosm headspace equilibrates with the downstream surfaces. Based on preceding tests using 20 ppm calibration gas, this time period was set to 30 min, including a 27 min equilibration time and a 3 min measurement period. The analytical devices comprised a cavity ring-down spectroscopy (CRDS) gas analyser (G2508, Picarro Inc., 3105, USA) for measuring NH_3, N_2O, CO_2, and CH_4 emissions, and a chemiluminescence detector (CLD 88 p, Eco Physics AG, Duernten, Switzerland) to monitor NO concentration. Mass flow meters (MFMs) were used to record the flow rates towards the gas analysers and the overflow (MFMα and MFMβ in Fig. 2). Calibration of the spectrometers was performed with reference gases (N_2O: 361.94 ppb; CO_2: 396.45 ppm, CH_4: 2.168 ppm, NO: 4.35 ppm; Air Liquide GmbH, Germany) in synthetic air prior to the experiment. In case dilution of the calibration gases was necessary, a multi-gas calibration system (series 6100; Environics Inc., Tolland, CT, USA) was used.

2.6. Measurement of plant biomass

At the end of the measurement period, the above-ground biomass was harvested and the fresh weight was measured using a laboratory scale. Subsequently, the plant samples were placed in an oven at 60 °C for one week to achieve a constant dry weight, following the protocol of Khan et al. (2024). The plant biomass is given in supplementary information (Table S3).

2.7. Data processing and statistical analysis

Trace gas flux rates were calculated using R (Statistical Computing Platform, v3.6.3) and Microsoft Excel (Microsoft Corporation, 2019; Microsoft, Seattle, WA, USA) based on the determined mixing ratios of flushing air and headspace air as well as on dissolved NH_4^+ for the dynamic chamber approach and the acid traps, respectively. Cumulative fluxes for the entire experimental duration, were obtained by multiplying the flux rates with the duration between two measured points. These area-scaled emissions were summed to provide the total cumulative emissions [kg ha^{-1}]. Regarding statistical analysis, the experimental design of independent experiments allows the determination of significant differences between slurry and slurry + amendment groups of the same experiment. For this, the Shapiro-Wilk test was conducted for normality tests, followed by one-way ANOVA (post-hoc multiple comparisons, Tukey's-b) for normally distributed data, while Kruskal-Wallis one-way ANOVA was used for non-normally distributed data. The named tests were carried out with SPSS (ver 27.0, IBM Corp., Armonk, NY, USA), a significance level of 95 % was used. Relative reductions of cumulative trace gas emissions, can be compared across experiments as they are normalized through the variability caused by differences in slurry composition. However, direct statistical comparison of cumulative emission between experiments were not carried out. Relations of cumulative trace gas fluxes to experiment parameters were investigated using stepwise multiple linear regression analysis in R. The full set of explanatory variables comprised the added amount of organic total N, NH_4^+, organic substance, acidity (H^+), phosphate and volume added to soil-plant mesocosms. To ensure that multicollinearity between explanatory variables was at an acceptable level, explanatory variables with a high variance inflation factor (VIF >5) were stepwise removed. For the plant biomass data, ANOVA analysis was performed using R (R Core Team, 2022, ver. 4.4.2, R Foundation for Statistical Computing, Vienna, Austria) to evaluate the statistical significance among different treatments. Figures and designs were created using OriginPro 2020 (OriginLab Corporation, 2020; OriginLab Corporation, Northampton, Massachusetts) and CorelDRAW (X7, Corel Corporation, Ottawa, Canada), respectively.

3. Results and discussion

3.1. Effect of amendments on ammonia emission

For better comparison with existing literature, which make use of acid traps to determine NH_3 emissions, results of this method are presented and discussed in this section, and compared to the CRDS gas analyser results in section 3.7. Acid trap NH_3 measurements showed nearly zero fluxes during the pre-incubation (days −3 to −1, Fig. 3) for all experiments and treatments, and the control treatment consistently exhibited the lowest, close to zero, fluxes throughout the observation period in all three experiments (Fig. 3). On the day of fertilizer application, peak fluxes and emissions were observed for all slurry (S) treatments, with flux rates amounting to, 0.85, 5.1 and 7.3 kg N ha^{-1} day^{-1} (Fig. 3) for experiments 1, 2 and 3, respectively. For subsequent days, fluxes were consistently lower and dropped to zero level. Since three independent experiments were conducted with different slurry characteristics, cumulative NH_3 emissions of the different experiments can only be compared for the control treatments (C) which did not receive any fertilizer. Cumulative NH_3 emissions of treatments C were close to the detection limit and not significantly different, showing that measurements were not affected by differences in soil-plant mesocosms. The cumulative emissions for treatments S were 1, 7, and 9 kg N ha^{-1} for experiments 1, 2, and 3, respectively (Fig. 3d–f). The stepwise multiple linear regression analysis (Table 2) showed that the loads of NH_4^+, H^+ and organic matter significantly control cumulative NH_3 emissions, which agrees with the literature (Sommer and Hutchings, 2001; Behera et al., 2013; Aguirre-Villegas et al., 2024). Since the standardized regression coefficient for NH_4^+ was highest, the emission level of the different experiments was controlled by the amount of NH_4^+ added to soil-plant mesocosms, with increasing loads of H^+ and organic matter content reducing the emissions. While the mechanism behind the reduction of NH_3 emission due to organic matter is an indirect effect to the correlation of organic matter and organic N ($r^2 = 0.44$), associated with a decrease of the ratio of NH_4^+ to total N except for SW ($r^2 = 0.85$), the effect of acidification is straightforward. Thus, amending slurry with cheese whey (SW) and slurry with sauerkraut juice (SSJ) significantly reduced NH_3 emissions by 91 % and 92 % (Fig. 3d and e), respectively, compared to their respective slurry treatments. These findings align with previous research, including studies by Pain et al. (1987) and Gioelli et al. (2022), as well as a meta-analysis by Emmerling et al. (2020), which reported NH_3 emission reductions in acidified slurries with H_2SO_4 ranging from 64 % to 95 %. Additionally, reductions of 68 % (Prado et al., 2020) and 67 % (Kavanagh et al., 2021) were observed using

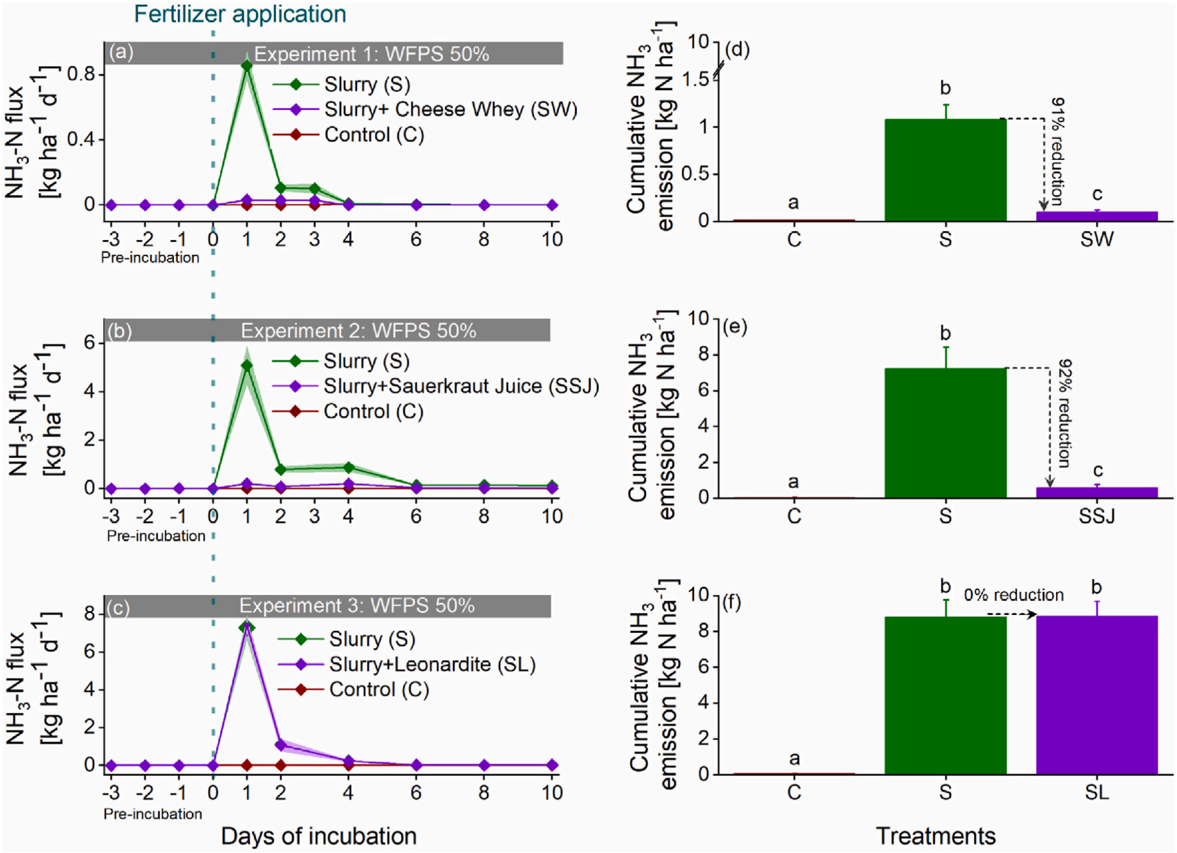

Fig. 3. Daily ammonia (NH_3-N) fluxes (a, b, c) and cumulative emission (d, e, f) of experiment 1, 2, and 3, respectively, were determined using acid traps. A pre-incubation period of 3 days preceded fertilizer application on day 0 (teal dashed line). Treatments control (C), slurry (S), slurry + whey (SW), slurry + sauerkraut juice (SSJ) and slurry + leonardite (SL) are shown in brown, green and violet color, respectively. Colored symbols with shades and bars with whiskers represent mean daily fluxes and cumulative emissions with their standard errors, respectively. Small letters (a, b, and c) show significant differences between treatments. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

Table 2
Stepwise multiple linear regression analysis of the effects of organic matter, ammonium, and hydrogen ion (H^+) loads on cumulative trace gas emissions (NH_3, N_2O and CO_2), including standardized regression coefficients (beta), and coefficients of determination (R^2 and adjusted R^2), and p-values. Data for NH_3 and N_2O were log-transformed, while CO_2 data were not log-transformed. The predictors included in this regression analysis (loads of organic matter, NH_4^+, and H^+) were selected based on their Variance Inflation Factor (VIF) values. A threshold of VIF <5 was used to ensure that multicollinearity among the predictors was at an acceptable level.

	NH_3		N_2O		CO_2	
Predictor	Estimates	Beta	Estimates	Beta	Estimates	Beta
(Intercept)	0.19 *		−0.01		−6.67	
Organic matter (g)	−0.53 ***	−0.31	−0.07 **	−0.39		
NH_4^+ (g)	42.25 ***	1.10	3.98 ***	1.01	1979.43 ***	0.69
H^+ (moles)	−3.9E+07 ***	−0.20	5.6E+06 **	0.28	4.2E+09 **	0.30
R^2/adj. R^2	0.91/0.90		0.70/0.68		0.60/0.56	

*p < 0.05; **p < 0.01; ***p < 0.001.

bio-acidification products such as cheese whey and brown sugar. To our knowledge, this is one of the first studies showing the NH_3 emission reduction potential of these biological waste products when used as slurry amendments and applied to soil-plant systems. Despite the practical limitations regarding the amount of amendment that needs to be added to slurry, the findings demonstrate that cheese whey and sauerkraut juice, when used within these constraints, exhibit a reduction potential comparable to that of synthetic sulfuric acid (H_2SO_4).

In contrast, amendment of slurry with leonardite (Fig. 3f) did not significantly reduce NH_3 emissions compared to their respective slurry treatments. This is in apparent contradiction to Cao et al. (2022) who observed a reduction in NH_3 emissions of 32–64 % for the addition of leonardite to cattle slurry, but also in this study the differences were not significant since the emission level was low compared to the variability among replicates. Cao et al. (2022) attributed the reduction of NH_3 emissions to the acidic character and adsorption of NH_4^+ to cation exchange sites of leonardite, indicating that the amount of leonardite added to the slurry plays a significant role. While in the named study approx. 108 g leonardite per liter of slurry were added, in our study only 20 g leonardite per liter were added. Though the level of application of this study is within the range of the 10–30 g l^{-1} recommended by the retailer, the amount chosen in our study may not have been sufficient to reduce the pH and adsorb enough NH_4^+ to decrease NH_3 emissions. This is supported by the higher pH of the mixture of slurry and leonardite in our study (pH 7, Table 1) compared to Cao et al. (2022) who reached a pH of 6.7 and 6.8. In addition, and probably more important, the slurry was incorporated into the top 2 cm of soil in the study by Cao et al. (2022). Even the shallow incorporation may have reduced the interface area of slurry and atmosphere and simultaneously amplified adsorption of slurry NH_4^+ to cation exchange sites in the soil. Consequently, our aim to reflect a fertilization of a winter crop, which prevents incorporation due to the presence of ryegrass plants, may have better isolated the amendment effect from the incorporation effect. Additionally, Cao et al. (2022) investigated two application techniques, i.e., sequentially adding leonardite and slurry to the soil, and mixing slurry and leonardite the day before application and observed highest NH_3 emission reduction (64 %) for application of slurry mixed with leonardite overnight, which suggests that some time is required for the impact of leonardite on NH_3 emission to take effect. As a result, the preparation of the SL slurry approximately 3 h before application may have contributed to the lack of NH_3 emission reduction in our study.

3.2. Effect of amendments on methane emissions

Methane fluxes ranged from −0.01 to 0.01 mg m^{-2} h^{-1} during pre-incubation for all treatments and during the entire observation period for all control treatments (Fig. 4a–c). The observation of low fluxes agrees with the literature since arable soils are usually small CH_4 sinks (Hansen et al., 2024) with the net CH_4 flux being the result of simultaneous microbial CH_4 consumption and production through methanotrophs and methanogens in aerobic and anaerobic soil compartments (Conrad et al., 1995; Laanbroek, 2010). Directly after fertilizer application, massive CH_4 fluxes were observed for the slurry treatment (S), which ranged up to 3.2, 2.7 and 1 mg m^{-2} h^{-1} for experiments 1, 2 and 3, respectively (Fig. 4a–c). These emissions rapidly declined for experiments 2 and 3 on the day of application, while they persisted to the day after application for experiment 1. The amendment treatments whey (SW), sauerkraut juice (SSJ) and leonardite (SL) showed lower CH_4 fluxes with peak emissions amounting to 0.2, 0.02, and 0.05 mg m^{-2} h^{-1} for experiment 1, 2 and 3, respectively. As for NH_3 emissions, there was no significant difference in cumulative CH_4 emissions between the control (C) treatments of experiments 1 to 3, showing that measurements were not affected by soil properties or differences in microbial activity.

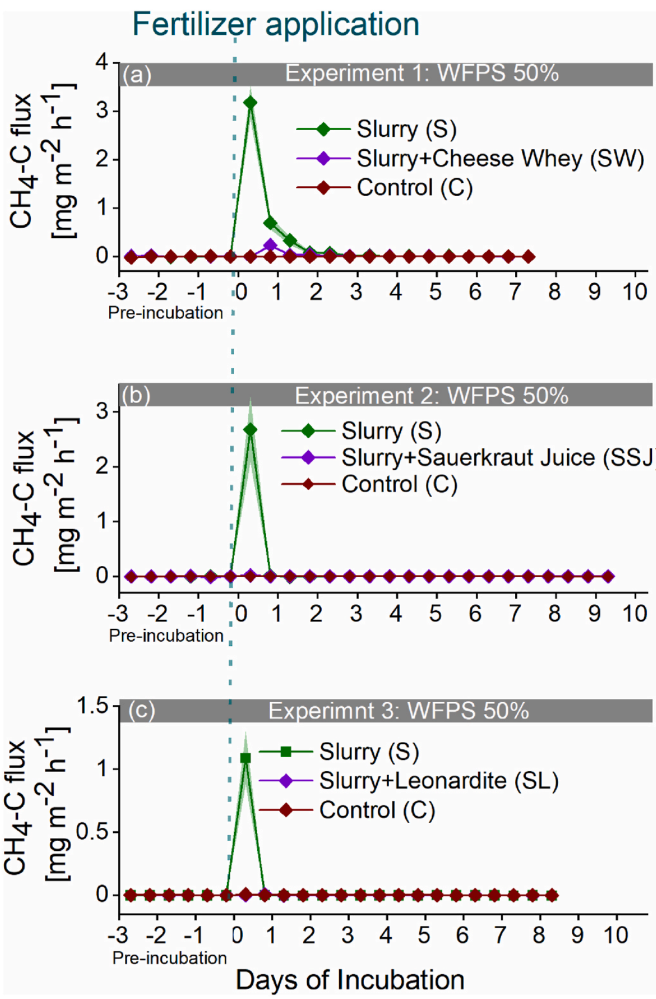

Fig. 4. Daily methane (CH_4-C) fluxes (a, b, c) of experiment 1, 2, and 3, respectively. A pre-incubation period of 3 days preceded fertilizer application on day 0 (teal dashed line). Treatments control (C), slurry (S), slurry + whey (SW), slurry + sauerkraut juice (SSJ) and slurry + leonardite (SL) are shown in brown, green and violet color, respectively. Colored symbols with shades mean daily fluxes with their standard errors, respectively. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

Pronounced CH_4 emissions have been observed immediately after fertilizer application, with rapidly decaying CH_4 flux rates explained by the release of CH_4 that was produced and dissolved in the slurry during storage. Lower fluxes which remained elevated for approx. five days on the other hand were related to CH_4 production in the soil (Sherlock et al., 2002; Dittert et al., 2005). The rapidly decaying CH_4 emissions in our experiments were similar to those of the study by Dittert et al. (2005), indicating that the observed CH_4 burst was due to dissolved CH_4. Release of dissolved CH_4 may be amplified by stirring. Since the mixture of slurry and amendment was additionally stirred in the preparation process, the preparation of the mixtures may have released significant amounts of CH_4 prior to application and could have contributed to the low emissions for the slurry-amendment mixtures. For this reason, cumulative CH_4 emissions were not calculated for the slurry treatments. However, previous studies on bio-acidification revealed, that the mixture of cheese whey to slurry notably decreased CH_4 emissions during storage experiments compared to pure slurry treatment (Berg et al., 2006; Wheeler et al., 2010; Samer et al., 2014; Prado et al., 2020). This reduction was primarily due to acidification, which lowers the pH of the slurry-amendment mixture, alters microbial activity and inhibits methanogenesis (Samer et al., 2014; Prado et al., 2020; Kavanagh et al., 2021; Gioelli et al., 2022; Regueiro et al., 2022). To our knowledge, there is no literature on sauerkraut juice (SSJ) used as a bio-acidification product, but since the CH_4 fluxes for SSJ were much lower than for the pure slurry the same mechanism for the reduction may have contributed to the lower emissions. There is also a scarcity of reference studies for our group SL in experiment 3 which investigates the impact of leonardite on GHG emissions after the application to soil-plant systems. Das et al. (2023) and Lee et al. (2024) observed that humic substances facilitate CH_4 adsorption and sequestration due to their high surface areas, so that the humic substances in leonardite may have contributed to the lower fluxes of CH_4 emissions (Fig. 4c). Additionally, the high organic matter content of leonardite may have enhanced soil microbial activity, particularly methane-oxidizing bacteria, converting CH_4 into less harmful compounds like CO_2 and H_2O (Bastami et al., 2016; Sarıyıldız, 2020; Wang et al., 2020; Piri et al., 2023).

3.3. Effect of amendments on carbon dioxide emissions

In soil-plant mesocosms, CO_2 is produced by plant and microbial respiration and consumed by plants during photosynthesis (Yankelzon et al., 2024). During the pre-incubation phase, CO_2 fluxes of all treatments ranged from −150 to 170 mg m^{-2} h^{-1}, and from −132 to 661 mg m^{-2} h^{-1} across all experiments during whole incubation period. The control treatment (C) consistently exhibited low flux variations throughout the experiment duration compared to slurry and amended treatments (Fig. 5a–c). Following fertiliser application, CO_2 fluxes temporarily increased across slurry (S) and slurry with amendment treatments (SW, SSJ, and SL, Fig. 5a–c). The highest average CO_2 flux rate was observed on the day of fertilizer application for the treatment S in experiments 1, 2, and 3 which amounted to 308, 661 and 657 mg m^{-2} h^{-1}, respectively (Fig. 5a–c). However, the fluxes for the slurry + amendment treatments (SW, SSJ, SL) were not significantly different from treatment S. The sign of the cumulative CO_2 flux for the different soil-plant mesocosms varied from positive to negative for treatment C (standard deviations of Fig. 5d–f), due to the interplay of respiration and photosynthesis, but the average cumulative emission was close to zero

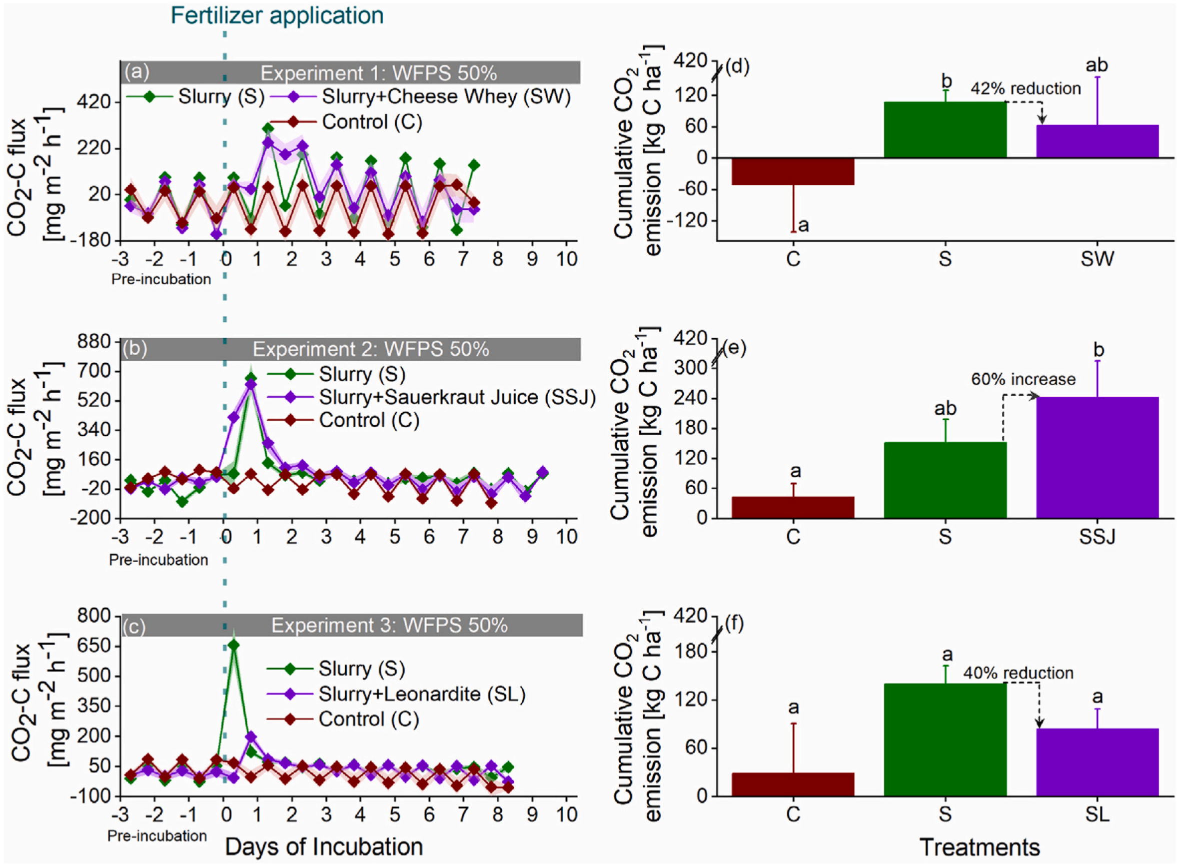

Fig. 5. Daily carbon dioxide (CO_2-C) fluxes (a, b, c) and cumulative emission (d, e, f) of experiment 1, 2, and 3, respectively. A pre-incubation period of 3 days preceded fertilizer application on day 0 (teal dashed line). Treatments control (C), slurry (S), slurry + whey (SW), slurry + sauerkraut juice (SSJ) and slurry + leonardite (SL) are shown in brown, green and violet color, respectively. Colored symbols with shades and bars with whiskers represent mean daily fluxes and cumulative emissions with their standard errors, respectively. Small letters (a, b, and c) show significant differences between treatments. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

for the entire monitoring period.

In contrast to all other trace gas emissions, cumulative CO_2 emissions between the control (C) treatments of experiments 1 to 3 were significantly different. However, as mentioned above, CO_2 fluxes are influenced by photosynthesis, plant respiration and microbial respiration. Thus, they don't exclusively reflect soil properties and microbial processes, so that we assume based on the other trace gas emissions that measurements were not affected by soil properties or differences in microbial activity. The cumulative CO_2 fluxes for the treatment S were 108, 152 and 140 kg C ha^{-1} (Fig. 5d–f), with the difference to the respective treatments C being significant only for SW. Similarly, the amended treatments of SW, SSJ and SL showed cumulative fluxes of 63, 242 and 84 kg C ha^{-1} in experiments 1 to 3, respectively, but these were not significantly different from the respective S treatments. Thus, taking the presence of plants into account, manure amendments did not have a significant effect on cumulative CO_2 emissions. This is supported by the stepwise multiple linear regression analysis in which organic matter load was eliminated in the stepwise process (Table 2). The most important predictor of CO_2 emissions was the NH_4^+ load in the fertilizer, indicating that the temporal increases in CO_2 emissions after fertilizer application were due to a short-term stimulation of soil microorganisms and plants, which increased their respiration. This means that the cumulative CO_2 emissions were dominated by photosynthesis and ecosystem respiration, and only to a small extent related to fertilization, so that the duration of the experiment arbitrarily determines the cumulative CO_2 emissions. The lack of significant effects on CO_2 emissions aligns with other studies by Prado et al. (2020) and Gioelli et al. (2022) who used cheese whey. Moreover, our study is consistent with other studies indicating that substances containing labile carbon can elevate CO_2 emissions directly after application or in the later phases but not significantly (Bastami et al., 2016; Prado et al., 2020; Kavanagh et al., 2021; Cao et al., 2022). Since leonardite is a source of labile carbon, it could increase C mineralization, however, the release of protons from acidic leonardite in soils can counteract this effect, potentially reducing CO_2 emissions (Tran et al., 2015; Cao et al., 2022).

3.4. Effect of amendments on nitrous oxide emissions

Nitrous oxide fluxes in the pre-incubation measurement days were close to the detection limit and fluctuated around 0–0.01 mg m^{-2} h^{-1} for all treatments, and for treatment control (C) throughout the entire experimental period (Fig. 6a–c). Following fertiliser application, fluxes increased for the slurry and slurry plus amendment treatments, but the magnitude of the fluxes and the duration of the increase was variable. The peaks of N_2O emissions observed in the two days after application likely result from both nitrification and denitrification. While acidification is known to delay nitrification (Sørensen and Eriksen, 2009; Fangueiro et al., 2013; Prado et al., 2020), the availability of NH_4^+ can still trigger nitrification, leading to N_2O and NO emissions. Simultaneously, anaerobic conditions and nitrate accumulation may promote denitrification, further contributing to N_2O release (Berg et al., 2006). Given these dynamics, both processes likely played a role in the observed emissions, highlighting the complex interactions between slurry acidification and N transformations in soil. Moreover, highest fluxes were observed for treatment S of experiment 3 (2.7 mg m^{-2} h^{-1}, Fig. 6c), followed by treatment SSJ (1.7 mg m^{-2} h^{-1}, Fig. 6b) of

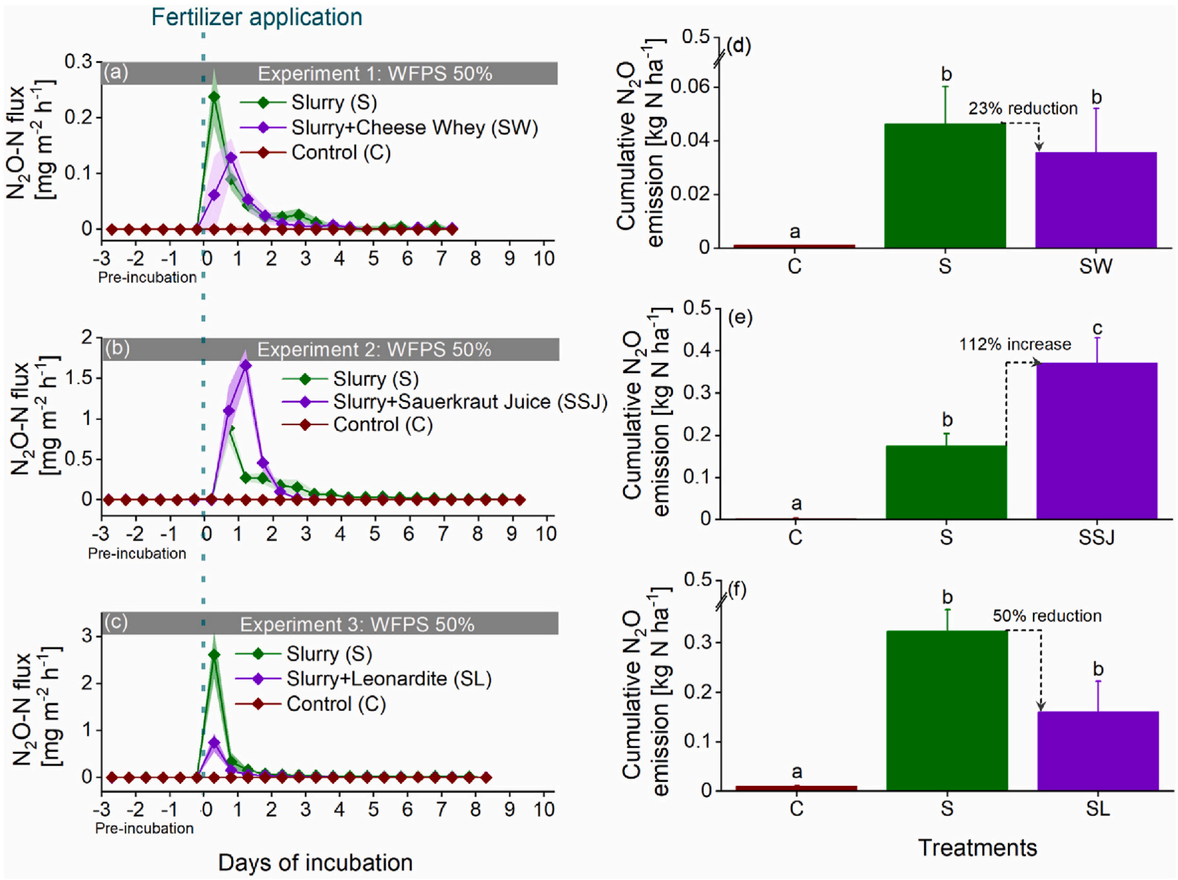

Fig. 6. Daily nitrous oxide (N_2O-N) fluxes (a, b, c) and cumulative emission (d, e, f) of experiment 1, 2, and 3, respectively. A pre-incubation period of 3 days preceded fertilizer application on day 0 (teal dashed line). Treatments control (C), slurry (S), slurry + whey (SW), slurry + sauerkraut juice (SSJ) and slurry + leonardite (SL) are shown in brown, green and violet color, respectively. Colored symbols with shades and bars with whiskers represent mean daily fluxes and cumulative emissions with their standard errors, respectively. Small letters (a, b, and c) show significant differences between treatments. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

experiment 2, treatment S of experiment 2 (0.8 mg m^{-2} h^{-1}, Fig. 6a) and treatment S of experiment 1 (0.25 mg m^{-2} h^{-1}, Fig. 6a). Also, for N_2O, cumulative emissions of the C treatments of experiments 1 to 3 were not significantly different, showing that the soil samples or microbial activity did not affect the measurements. The cumulative N_2O emission of treatment SW was by 23 % lower than that of treatment slurry (Fig. 6d), with the difference being not significant. At first glance, this is surprising since the efficient reduction of NH_3 emissions compared to treatment S of the SW treatment must have increased the availability of NH_4^+ in the soil for nitrification and subsequent denitrification, both of which can produce N_2O. On the one hand, low N_2O emissions in the SW experiment may indicate that soil conditions, specifically soil water content, were not conducive for denitrification. However, the amount of liquid that was added as slurry-whey mixture was 20 % higher for SW compared to treatment S. This led to increased water content of SW mesocosms, which stimulates denitrification and N_2O emissions. Thus, water content cannot be solely responsible for the lower N_2O emissions in experiment 1, since other treatments at the same initial moisture level (50 % WFPS) showed higher emissions. For this reason, it seems like the lower ratio of NH_4^+ to total N content of the slurry and slurry-whey mixture in experiment 1 contributed to the low N_2O emission level compared to the other experiments. Within experiment 1, the tendency of reduced N_2O emission due to whey addition agrees with Samer et al. (2014) and Prado et al. (2020), who reported that N_2O emissions of slurry were reduced at decreased pH levels following cheese whey addition, so that a combination of the effect of NH_4^+ load and pH may have led to the observed insignificant difference in cumulative N_2O emission in experiment 1. This is supported by the results of the stepwise multiple linear regression analysis. Since the standardized regression coefficient was positive and highest for NH_4^+ load, this parameter dominates the emission level while the negative coefficient for H^+ is in line with the literature (Samer et al., 2014; Prado et al., 2020) and our observation of lower N_2O emissions at the lower slurry pH of experiment 1. At the same time, the slurry with higher organic content had a lower NH_4^+ to total N ratio, so that the slurry with highest organic content of experiment 1 resulted in the lowest N_2O emissions. In addition, other products like sugar, citrus juice, and orange juice were more effective in reducing N_2O emissions compared to cheese whey (Samer et al., 2014; Prado et al., 2020). The weaker capability of cheese whey in reducing N_2O emissions compared to other products might be due to NH_3 formation during cheese whey protein hydrolysis. This NH_3 undergoes nitrification, leading to the formation of NO_3^-, which can then be denitrified to N_2O (Gioelli et al., 2022).

Similarly, cumulative N_2O emission of the leonardite treatment was 50 % lower than that of S (Fig. 6f), but the difference was not significant. This observation agrees with the study of Cao et al. (2022), who reported that leonardite decreases N_2O emissions by adsorbing NH_4^+ of the slurry on cation exchange sites and in the process reducing the amount of substrate for nitrification and subsequent denitrification, the main processes causing N_2O emissions. However, the much lower amount of leonardite added to the slurry in this study may have rendered this reducing effect on N_2O production insignificant.

In contrast to experiments 1 and 3, significantly higher cumulative N_2O fluxes were observed for the slurry amendment with sauerkraut juice (Fig. 6e). Based on the stepwise multiple linear regression analysis (Table 2), the lower pH and NH_4^+ load of treatment SSJ compared to treatment S should result in a lower cumulative emission of SSJ compared to S. However, the efficient reduction of NH_3 emissions through sauerkraut juice, results in a higher availability of NH_4^+ in the SSJ soil-plant mesocosms compared to those of S. While the same situation (higher NH_4^+ due to lower NH_3 emission) was met in experiment 1, where the pH-effect dominated and reduced N_2O emissions for SW compared to S, the NH_4^+ load was higher in experiment 2. Consequently, in total more substrate was accessible to nitrifying and denitrifying microorganisms capable of producing N_2O in experiment 2, which overruled the pH-effect. Thus, our findings are consistent with those of other studies (Velthof et al., 2003; Van Nguyen et al., 2017; Cao et al., 2022; Wang et al., 2023; Li et al., 2024) that observed emissions of N_2O following higher NH_4^+ availability through fertilizer application and acid amendment applications.

In particular, the different N_2O flux levels across the experiments suggest that SW and SSJ effectively reduced NH_3 emissions. However, the effect of such measures on N_2O emissions will ultimately depend on soil conditions at the point in time of application and the mineral N content of the fertilizer, so that amendments with a low NH_4^+ to total N content ratio are preferable.

3.5. Effects of amended treatments on nitric oxide

In soil-plant mesocosms, NO is produced through microbial nitrification and denitrification. While nitric oxide itself is not a GHG, it acts as a precursor to the formation of N_2O, amplifying its impact on climate change (Butterbach-Bahl et al., 2011). Nitric oxide fluxes exhibited a variable pattern across all treatments throughout the experiments and consistently were in the range of −0.01 to 0.28 mg m^{-2} h^{-1} (Fig. 7a–c). During the pre-incubation measurement, fluxes ranged from −0.005 to 0.014 mg m^{-2} h^{-1} across all treatments. In the control treatment, negative values were occasionally observed throughout the experiments. Following fertiliser application, fluxes increased slightly, with treatment SSJ in experiment 2 showing a value of 0.28 mg m^{-2} h^{-1} after fertilizer application (Fig. 7b). The other amended treatments of SW and SL showed a comparable temporal flux trend to their respective treatment S (Fig. 7a–c). Since flux rates did not return to the levels observed for the control treatment until the end of the experiment, cumulative NO fluxes were not calculated. Pilegaard (2013) and Wu et al. (2010) found that nitrification is the primary source of NO emissions at low WFPS, while in wet soils, denitrification prevails, consuming much of the NO before it can be released into the atmosphere and favouring N_2O emission. In our study, it is likely that manure application temporarily increased soil moisture content to a level conducive to N_2O production. As the experiment progressed, seepage distributed water and nutrients from the slurry throughout the soil, which may have resulted in soil moisture levels more favourable for NO production due to the nitrification after N_2O emissions had peaked. This suggests that future studies on NO emissions should consider a longer experiment duration.

3.6. Ammonia pollution swapping on nitrous oxide and nitric oxide

One of the major concerns regarding (bio-) acidification of slurry is that NH_3 reduction could lead to increased emissions of N_2O and NO. However, our study found that amending slurry with cheese whey significantly reduced NH_3 emissions and also decreased N_2O and NO emissions, consistent with the findings by Prado et al. (2020) and a meta-analysis by Emmerling et al. (2020). In contrast, the amendment treatment of slurry with sauerkraut juice significantly decreased NH_3 emissions but resulted in higher N_2O and NO emissions. Following the application of sauerkraut juice to soil-plant mesocosms, N_2O and NO fluxes remained lower and comparable to those from pure slurry treatment, suggesting that the increase in emissions could be attributed to soil nitrification and denitrification processes influenced by substrate availability and pH, in agreement with studies by Cao et al. (2022), Wang et al. (2023), and Li et al. (2024). Conversely, leonardite did not reduce NH_3 emissions but also decreased N_2O (50 %) and NO emissions (22 %), aligning with the findings of Cao et al. (2022).

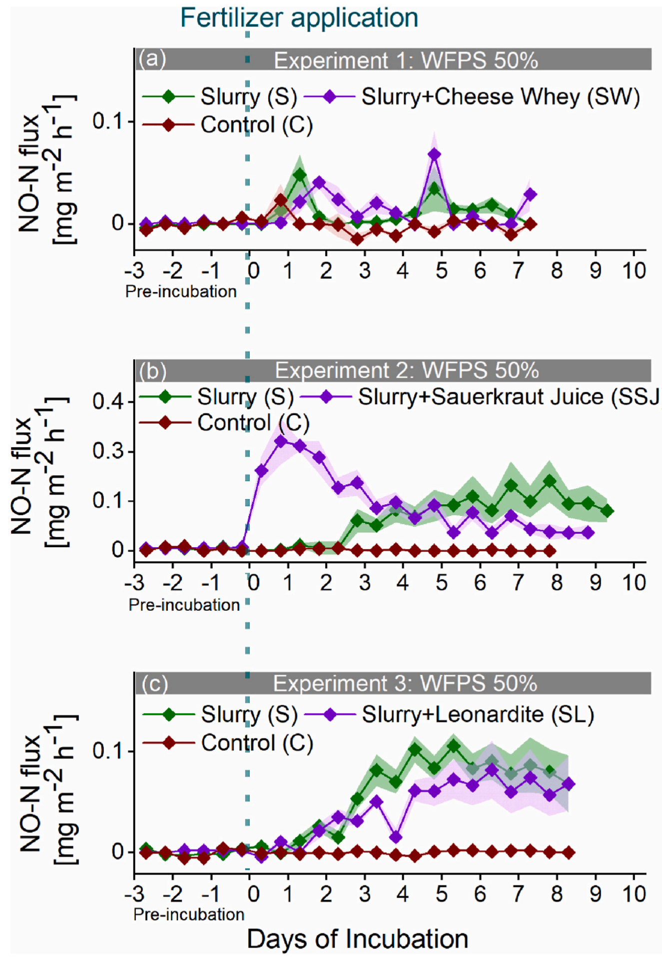

Fig. 7. Daily nitric oxide (NO-N) fluxes (a, b, c) of experiment 1, 2, and 3, respectively. A pre-incubation period of 3 days preceded fertilizer application on day 0 (teal dashed line). Treatments control (C), slurry (S), slurry + whey (SW), slurry + sauerkraut juice (SSJ) and slurry + leonardite (SL) are shown in brown, green and violet color, respectively. Colored symbols with shades represent mean daily fluxes with their standard errors, respectively. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

3.7. Discrepancy of ammonia emission by different approaches

Ammonia emissions measured by the CRDS gas analyser ranged from −0.01 to 0.02 mg m^2 h^{-1} during the pre-incubation days, with the control treatment consistently exhibiting the lowest fluxes throughout the experiment (Fig. 8a–c). On the day of fertilizer application, peak NH_3 fluxes were observed from the slurry treatment (S) in experiment 3 (92 mg m^{-2} h^{-1}, Fig. 8c), followed by experiment 2 (53 mg m^{-2} h^{-1}, Fig. 8b) and experiment 1 (16 mg m^{-2} h^{-1}, Fig. 8a). Unlike NH_3 fluxes measured using the acid trap approach, the high fluxes were increased for several days after fertilizer application (3–5 days, Fig. 8a–c) which led to differences in cumulative NH_3 emission. In agreement with the acid traps, amended treatment fluxes were also significantly lower using the CRDS gas analyser, and showed comparable NH_3 reductions of 90 % by cheese whey and sauerkraut juice, and 42 % reduction by leonardite (Fig. 8d–f).

The cumulative NH_3 emission by the CRDS gas analyser for treatment S in experiments 1, 2 and 3 were 4.17, 12.15 and 17.22 kg N ha^{-1}

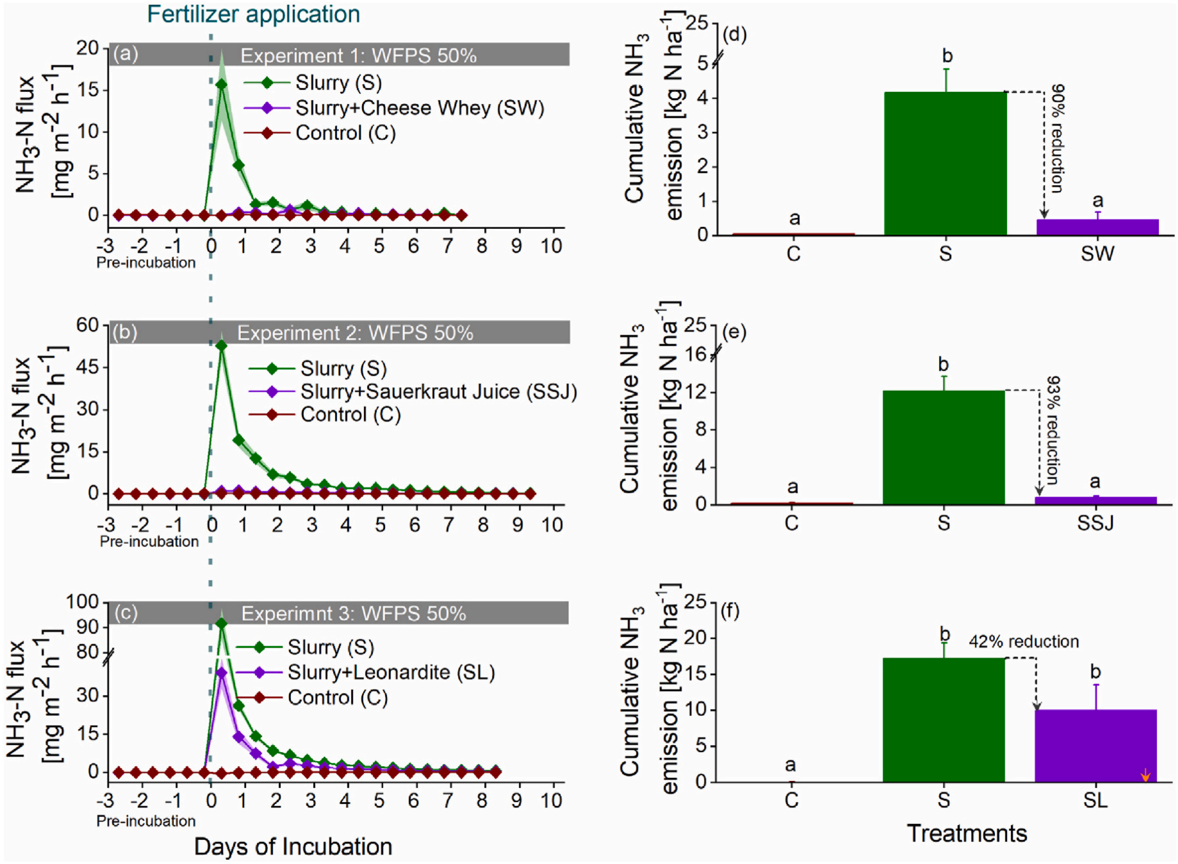

Fig. 8. Daily ammonia (NH_3-N) fluxes (a, b, c) and cumulative emission (d, e, f) of experiment 1, 2, and 3 respectively. A pre-incubation period of 3 days preceded fertilizer application on day 0 (teal dashed line). Treatments control (C), slurry (S), slurry + whey (SW), slurry + sauerkraut juice (SSJ) and slurry + leonardite (SL) are shown in brown, green and violet color, respectively. Colored symbols with shades and bars with whiskers represent mean daily fluxes and cumulative emissions with their standard errors, respectively. Small letters (a, b, and c) show significant differences between treatments. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

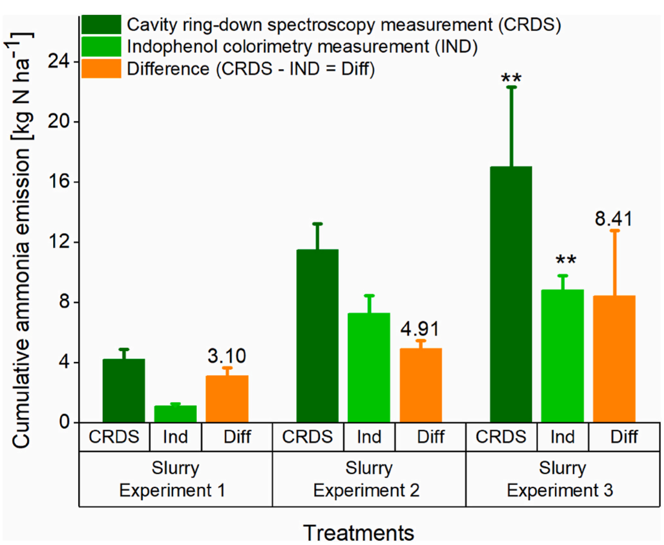

Fig. 9. Cumulative ammonia (NH_3) fluxes of slurry (S) treatments from experiments 1, 2 and 3. The green and light green color represent the measurement collected by the CRDS gas analyser and indophenol colorimetry (IND), respectively, while the differences are shown in orange. Asterisk show the significant difference (**: $p < 0.01$). (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

(Fig. 9), respectively, while the corresponding acid trap cumulative emissions were, 1.08, 7.24 and 8.81 kg N ha^{-1}, respectively. Consequently, the difference was approximately 3.10, 4.91, and 8.41 kg N ha^{-1} higher for the CRDS gas analyser in experiments 1, 2, and 3, respectively.

Underestimation of acid traps may arise if the acid liquid saturates with NH_4^+. However, Janz et al. (2021), calculated that 100 ml of oxalic acid is sufficient for trapping an amount equivalent to 200 kg NH_4^+-N ha^{-1} for the mesocosms used for this study. Considering that in the current study, the fertilizer application rate was 80 kg N ha^{-1}, with NH_4^+, the direct NH_3 precursor, being only a fraction of this, and that the trap solution was exchanged every day, a saturation of the acid traps appears unlikely. Furthermore, the airflow (0.6 L min^{-1}) through the oxalic acid traps, height, and amount of oxalic acid were aligned with recommendations of a previous study to accurately capture all NH_3 emissions over a 24-h period (Ndegwa et al., 2009). However, this alignment was designed to resolve the expected peak emissions, but the same study had shown that the sensitivity of acid traps operated at the same flow rate decreased with NH_3 concentration. Consequently, reduction of the flushing gas flow rate might have led to a more accurate result of the acid traps, but the accuracy gain reported for a reduction of flow rate by 50 % was in the range of 4 %, so that this cannot be the only reason for the differences between the measurement approaches (Ndegwa et al., 2009).

In contrast, the higher values recorded by the CRDS gas analyser could be attributed to several factors. First, the sampling resolution of the CRDS gas analyser was 30 min per soil-plant mesocosm, with each treatment (a set of six mesocosms) measured for 3 h in a 12-h cycle, whereas the gas washing bottle of oxalic acid traps sampled continuously for 11.30 h, so that the rapid decline of NH_3 fluxes was probably not adequately captured by the CRDS system. Second, the CRDS gas analyser (G2508, Picarro Inc., 3105, USA) used in this experiment allows for automatic correction of NH_3 up to 2 ppm, according to the manufacturer, but most of the NH_3 concentrations after slurry application exceeded this range. However, the linearity of the analyser was tested prior to the experiments and showed linearity up to a concentration of 20 ppm. Though the reason for the large differences between these two measurement approaches could not be resolved in this study, one major complication of the use of this specific CRDS gas analyser is the slow response time, and the long flushing times required to accurately separate different treatments in which NH_3 concentrations differ by several orders of magnitude. However, if used in settings that require observation of single treatments, the high temporal resolution of measurements may be beneficial.

3.8. Cost-effectiveness

In this experiment, we investigated three materials: cheese whey, sauerkraut juice, and leonardite. Cheese whey and sauerkraut juice have no economic value in Germany, so that they are available at no cost. However, their transport causes costs so that their use may only be appealing for farms close to the locations of dairy plants or canned food producers. Since whey and sauerkraut juice need to be transported away from their production sites anyway, utilizing these products in the vicinity of their production sites may reduce GHG emissions due to their transport and using them to acidify slurry represents a further step towards closed nutrient cycles with the additional benefit of reducing NH_3 emissions. In addition, it may allow producers to reduce the costs associated with treating and transporting these materials to landfills or draining after filtration.

In contrast, leonardite costs between ∼0.2 and 2 Euros per kg, depending on quality, quantity purchased and packaging size. At an application rate of 2 % mass, costs would amount to 4–40 € per m^3 slurry. However, this monetary valuation does not consider the energy (and GHG) investment for extraction and potential CO_2 release of leonardite degradation and decomposition (Punia, 2021).

3.9. Practical implication

Experiments 1 and 2 showed that cheese whey and sauerkraut juice significantly reduced ammonia emission. Although these additives show promise from a NH_3 reduction perspective, their practical adoption will depend on proven environmental benefits, agronomic benefits, farmers' willingness to integrate them, and market demand. At this stage, widespread use cannot be recommended without further assessment. To fully understand their effectiveness and implications of their use, it is important to investigate the effects of different slurry and amendment compositions and to conduct field studies assessing their impact on soil health and crop yields. Specifically, for sauerkraut juice, the impact of the high salt content and the effect on emissions during storage need to be investigated (Table S2). Additionally, it is crucial to note that the 1:2 application ratio alters the mixture from traditional animal slurry to a blend of organic materials. This may have legal implications, as regulations can vary between animal manure, other organic materials and countries. Therefore, further research is needed to validate the effectiveness and long-term impacts of these amendments on soil health and agricultural productivity, as well as to ensure compliance with legal standards.

4. Conclusion

This study explores the impact of incorporating cheese whey, sauerkraut juice, and leonardite into slurry on the release of trace gases emissions when applied to soil-plant mesocosms using an automatic soil incubation system. Cheese whey significantly reduced emission of ammonia (91 %). Sauerkraut juice, significantly reduced ammonia (92 %) but significantly increased nitrous oxide emissions (112 %). Leonardite reduced ammonia (42 %) and nitrous oxide (50 %) emission but not significantly. However, the application rate of leonardite remains ambiguous due to the absence of a definitive criterion, underscoring the necessity for further research in this area. Cheese whey and sauerkraut juice show higher potential for reducing ammonia emissions and advancing a recycling economy, but the effect of different slurry and amendment compositions as well as field studies including effects on yields are required. Regarding the ammonia measurement, the slow response time of the gas analyser reduces the temporal resolution of measurements when used in automatic incubation systems, making the acid trap approach preferable for comparing several treatments simultaneously.

CRediT authorship contribution statement

Fawad Khan: Writing – original draft, Formal analysis, Data curation, Conceptualization. **Samuel Franco-Luesma:** Writing – review & editing, Methodology, Investigation, Formal analysis, Data curation. **Baldur Janz:** Writing – review & editing, Methodology, Investigation, Data curation. **Michael Dannenmann:** Writing – review & editing, Supervision, Formal analysis. **Rainer Gasche:** Writing – review & editing, Methodology. **Andreas Gattinger:** Writing – review & editing, Supervision, Conceptualization. **Waqas Qasim:** Writing – review & editing, Formal analysis. **Ralf Kiese:** Writing – review & editing, Supervision, Conceptualization. **Benjamin Wolf:** Writing – review & editing, Supervision, Project administration, Methodology, Funding acquisition, Formal analysis, Conceptualization.

Declaration of competing interest

The authors have no relevant financial or non-financial interests or competing interests to disclose. They certify no affiliations with organizations having financial or non-financial interests in the subject matter. They also declare no financial or proprietary interests in any material discussed.

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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.2025.145753.

Data availability

Data will be made available on request.

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