

## Article

# Reduction of NH<sub>3</sub> Emissions from Urea by Long-Term No-Tillage and Stover Mulching with Inhibitors: An Isotopic Field Study in Northeast China

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**Abstract:** Large nitrogen (N) losses during fertilization in agricultural production may result in energy wastage, soil and water contamination, and potentially influence crop development. Thus, with the help of a <sup>15</sup>N-labeled tracer, we carried out a field monitoring analysis of NH<sub>3</sub> emissions in a long-term (9-year) conservation tillage agroecosystem of Mollisols in northeast China, in order to determine whether a no-tillage regime and four levels of stover mulching (0%, 33%, 67%, and 100%), combined with urease and nitrification inhibitors, could improve fertilizer utilization efficiency in agricultural systems by reducing ammonia volatilization. Our results showed that in comparison with ridge tillage, no-tillage with stover mulching levels of 33%, 67%, and 100% significantly reduced NH<sub>3</sub> emission rates and cumulative volatilization from 159.67 to 130.42 g N ha<sup>-1</sup> and <sup>15</sup>N-NH<sub>3</sub> cumulative volatilization emission by 26% (on average). Furthermore, the application of urease and nitrification inhibitors significantly reduced <sup>15</sup>N-NH<sub>3</sub> volatilization levels from 1.19 to 0.98 g N ha<sup>-1</sup>. Our research results demonstrate that a long-term no-tillage regime and straw mulching can significantly reduce NH<sub>3</sub> volatilization in fertilizers. Furthermore, when combined with the use of urease and nitrification inhibitors, these practices further enhance the reduction in NH<sub>3</sub> volatilization. Although the volatilization of <sup>15</sup>N-NH<sub>3</sub> is minimally studied in Mollisols, these findings provide a solid foundation for improving fertilizer utilization efficiency, reducing crop production costs and mitigating subsequent environmental pollution.

**Keywords:** no-tillage; stover mulching; NH<sub>3</sub>; <sup>15</sup>N-labeled tracer; nitrification inhibitors



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## 1. Introduction

In recent years, the world economy has faced many challenges, such as climate change and environmental pollution, resulting in global food security issues [1]. Nitrogen (N) fertilizer is one of the essential fertilizers in crop production, and the growing demand for food increases the use of N fertilizers. However, the improper application of N fertilizer can easily lead to the loss of N through NH<sub>3</sub> volatilization, N leaching, and runoff immediately following application [2], eventually resulting in an increasing loss of active N, such as through ammonia (NH<sub>3</sub>) volatilization to the environment, along with the corresponding adverse impacts [3]. Urea is the most widely traded N fertilizer in the world, and China accounts for 29% of its world-wide production [4]. It has been shown that the application of synthetic N fertilizer is one of the largest sources of NH<sub>3</sub> emissions in the world [5], and

has thus become the main N loss pathway in agricultural soils [6]. Furthermore, the low utilization rate associated with nitrogen fertilizer will further reduce agricultural productivity and seriously damage natural ecosystems [7,8].  $\text{NH}_3$  emission can indirectly lead to the acidification and eutrophication of soil and water systems and is an indirect source of the greenhouse gas  $\text{N}_2\text{O}$  [9–11].  $\text{NH}_3$  can react with  $\text{HNO}_3$  and  $\text{H}_2\text{SO}_4$  in the atmosphere to form secondary inorganic aerosols such as  $(\text{NH}_4)_2\text{SO}_4$ ,  $\text{NH}_4\text{NO}_3$ , and  $\text{NH}_4\text{HSO}_4$ . These aerosols account for an estimated 25–60% of China's overall  $\text{PM}_{2.5}$  levels [12–14]. Thus, as one of the key factors governing haze formation [15], a reduction in  $\text{NH}_3$  volatilization by 50% may lead to a reduction in  $\text{PM}_{2.5}$  by over 10% [16]. Therefore, considering the critical importance of reducing  $\text{NH}_3$  emissions, resulting from the application of fertilizer, the development of effective conservation management strategies is urgently needed to enhance the quantity and quality of agricultural production in sustainable agricultural systems.

Agricultural systems have the potential to benefit from conservation agriculture with sustainable management practices, which includes practices like crop residue mulching, minimally or entirely avoiding the disturbance of soil, and soil fertility improvement [17,18]. Previous studies showed that conservation tillage offers advantages over conventional tillage in terms of increasing soil organic carbon content [19], microbial biomass levels and diversity [20]. Crop residue mulching can affect soil biological, physical, thermal and hydrological properties [21,22]. For example, a no-tillage regime or straw mulching can improve the availability of plant nutrients through reducing the amount of N loss via leaching and runoff, and further increase crop yields [23,24]. However, researchers have reported that  $\text{NH}_3$  emissions under conservation tillage are higher than those under conventional tillage following the application of urea [25], because straw residues prevent urea from entering the soil and subsequently impair the effectiveness of urea fertilizer [26]. Additionally, urea exposed to soil is more readily hydrolyzed, resulting in increased urease activity [27]. In contrast, other studies have suggested that conservation tillage can significantly reduce  $\text{NH}_3$  emissions [28] affected by the degree of straw mulching [21]. Therefore, it is crucial to understand the impacts of tillage practices with different levels of straw mulching on  $\text{NH}_3$  volatilization, especially those derived from ammonium and urea N fertilizers, and find effective conservation management techniques to boost crop output and increase N use efficiency.

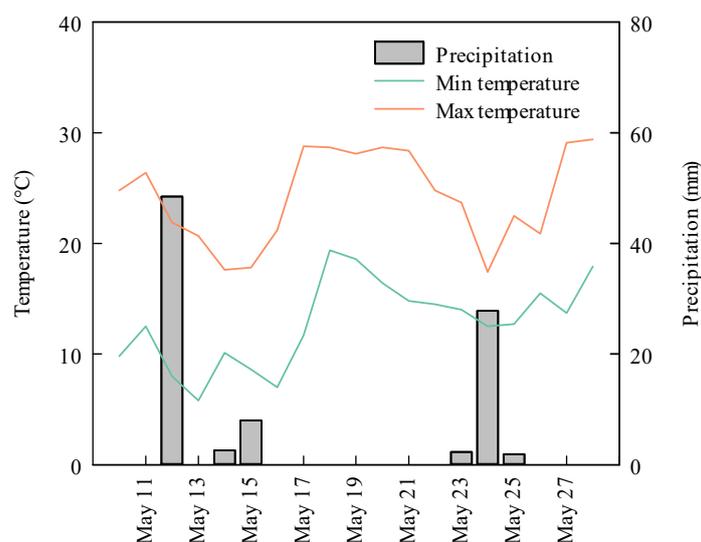
Urease inhibitors, for example N-(n-butyl) thiophosphoric triamide (NBPT), are widely used additives that stabilize urea by delaying its hydrolysis to  $\text{NH}_3$  and slow the microbial autotrophic oxidation of ammonium to nitrate [29,30], potentially leading to a reduction in N loss [31]. It was reported that urease inhibitors can reduce  $\text{NH}_3$  emission by up to 88% [32], while nitrification inhibitors, for example 3,4-Dimethyl-1H-pyrazolium dihydrogen phosphate (DMPP), can indirectly increase crop yields [33] due to their inhibitory effects on nitrification and denitrification processes [34]. Despite their potential adverse effects during application, studies have demonstrated that the benefits of nitrification inhibitors (NIs) outweigh the risks when considering all the relevant environmental and crop factors [33]. For instance, nitrification inhibitors may prolong the time that ammonium stays in the soil and increase the risks of  $\text{NH}_3$  volatilization [35,36]. Moreover, whether the concurrent application of urease and nitrification inhibitors can balance the loss of  $\text{NH}_3$  and nitrate to reduce the total N loss remains in question [37]. Studies have reported that the combined application of urease/nitrification inhibitors can reduce  $\text{NH}_3$  loss [38] and nitrate leaching [29], compared with a single application of one inhibitor. However, other studies have found that the combination of two inhibitors may also increase  $\text{NH}_3$  emissions [39–41]. Therefore, the high-resolution monitoring of  $\text{NH}_3$  emissions derived from urea is needed in relation to testing the impacts of the combined use of two inhibitors and other conservation management practices.

Northeast China is a major agricultural production area with respect to Mollisols, in which intensive agricultural management is potentially precipitating severe nutrient losses, indicating the need for sustainability in conservation management practices. Here, we present a long-term (9-year) continuous conservation management study conducted on a maize cropping system in Northeast China, in which  $^{15}\text{N}$ -labeled tracer techniques were used to investigate the impact of no-till farming and varying levels of maize stover mulching, in combination with urease and nitrification inhibitors, on  $\text{NH}_3$  volatilization from synthetic fertilizer N. Our previous studies showed that no-tillage farming and maize stover mulching with urease/nitrification inhibitors could improve N fertilizer retention in the studied Mollisols [42,43]. Thus, in this study, we hypothesized that: (1) applying maize stover mulching in no-till farming would significantly reduce  $\text{NH}_3$  volatilization from urea; and (2) the combined use of urease/nitrification inhibitors would further minimize the loss of urea to  $\text{NH}_3$ , leading to a lower N loss potential in conservation agricultural ecosystems.

## 2. Materials and Methods

### 2.1. Study Site

The study site, located in Lishu County, Siping City, Jilin Province ( $43^\circ 19' \text{ N}$ ,  $124^\circ 14' \text{ E}$ ), is characterized as a temperate region with a humid to semi-humid continental monsoon climate. Its annual mean temperature is  $6.9^\circ \text{ C}$  with a range of  $-13.5^\circ \text{ C}$  (January) to  $23.7^\circ \text{ C}$  (July). The changes in temperature and rainfall that occurred in the sampling site during the experiment are shown in Figure 1. This region receives an annual mean precipitation level of approximately 614 mm, about 75% of which occurs in the summer from June to September. The soil in the study area is classified as Mollisol, according to the U.S. Soil Taxonomy system. Table 1 presents the essential chemical properties of the top 20 cm soil layer in April of 2007, prior to the implementation of the no-till treatment. The prevailing agricultural practice in the study area is maize cultivation at a planting density of  $60,000 \text{ ha}^{-1}$ . After the harvesting of maize in autumn, no other crops were grown. To manage weeds and pests, herbicides were used annually for one week following sowing, while pesticides were applied during the 5–6 leaf and heading stages of maize using an automated high-stem dispenser.



**Figure 1.** Precipitation and environmental temperature variations in the analyzed area during study time from 11 May to 27 May in 2016.

**Table 1.** Physical and chemical properties of top 20 cm soil layer.

Physical Properties			Chemical Properties	
Texture (%)	Sand	28.5	Total organic C (g kg <sup>-1</sup> )	11.3
	Silt	38.6	Total N (g kg <sup>-1</sup> )	1.2
	Clay	32.9	Total P (g kg <sup>-1</sup> )	0.38
Clay Mineral (<2 µm, %)	Illite	14.5	Total K (g kg <sup>-1</sup> )	24.3
	Chlorite	30.0	Alkaline N (mg kg <sup>-1</sup> )	90.1
	Montmorillonite	24.2		
	Vermiculite	2.7	Avail. P (mg kg <sup>-1</sup> )	6.9
	Kaolinite	23.3	Avail. K (mg kg <sup>-1</sup> )	143.6
	Quartz	5.0	pH	7.1
	Feldspar	0.3		

## 2.2. Design of the Experiment

The present study reports on a long-term field experiment initiated in April of 2007, which was designed as a completely randomized block with four replicates. Each plot (replication) measured 261 m<sup>2</sup> (8.7 m × 30 m). The experiment comprised six treatments: (1) conventional ridge tillage (RT); (2) no-tillage farming with 0% maize stover mulching (NT0); (3) NT farming with 33% mulching (NT33); (4) NT farming with 67% mulching (NT67); (5) NT farming with 100% mulching (NT100); and (6) NT100 farming with 20% NPK reduction (NT100R). The no-till plots were left undisturbed, with the exception of maize planting during the spring season. After each harvest, maize stover was uniformly spread across the field's surface. A maize stover mulching rate of 100% was implemented within the plot, with a quantity of 7500 kg ha<sup>-1</sup>.

To accurately quantify the transformation and fate of fertilizer-derived N in the soil-crop systems, a <sup>15</sup>N-labeled micro-plot test was conducted from 2016 to the present within the 24 plots of above-mentioned field experiment. To do so, a micro-plot with an area of 4 m<sup>2</sup> (2 m × 2 m) was established in each plot. Meanwhile, an additional micro-plot of the same area was created in each NT100R plot for treatment with urease and nitrification inhibitors (NT100RI). The urease inhibitor employed was N-(n-Butyl) thiophosphoric triamide (NBPT), and the nitrification inhibitor used was 3,4-Dimethyl-1H-pyrazolium dihydrogen phosphate (DMPP). Subsequently, seven <sup>15</sup>N-labeled micro-plot test treatments were examined: (1) RT, (2) NT0, (3) NT33, (4) NT67, (5) NT100, (6) NT100R, and (7) NT100RI. Each treatment included four replicates, resulting in 28 micro-plots. Twenty-four maize plants were planted in each micro-plot on 10 May 2016, according to a plant density of 60,000 ha<sup>-1</sup>.

Similar application amounts and forms of synthetic fertilizers, including urea, concentrated super-phosphate, and KCl, were employed in the micro-plot test and the long-term field experiment. On 10 May 2016, urea fertilizer, labeled with a 9.80% <sup>15</sup>N abundance from Shanghai Research Institute of Chemical Industry, Shanghai, China, was applied in each micro-plot. Repeat measurements of NH<sub>3</sub> volatilization were then carried out daily from 11 May to 27 May 2016, amounting to a total of 17 days. In each micro-plot, synthetic fertilizers (basal fertilizers) were applied once a year at a soil depth of 10–15 cm, utilizing traditional NPK rates of 240 kg N ha<sup>-1</sup>, 110 kg P<sub>2</sub>O<sub>5</sub> ha<sup>-1</sup>, and 110 kg K<sub>2</sub>O ha<sup>-1</sup>. With the basal fertilizers, NBPT and DMPP were administered in every micro-plot at a rate equivalent to 1% of the urea fertilization rate.

## 2.3. Sampling and Analytical Methods

After applying fertilizer to agricultural soils, nitrification may convert NH<sub>4</sub><sup>+</sup>-N into highly mobile NO<sub>3</sub><sup>-</sup>-N within 2 weeks [41]. NH<sub>3</sub> emission was measured at 8:00 on days 1, 4, 5, 6, 7, 8, 10, 12, 14, and 17 after fertilization. The following equipment was utilized for the measurement of NH<sub>3</sub> volatilization: two sponges with a diameter of 16 cm and a thickness of 2 cm, which were soaked evenly in 15 mL of glycerol phosphate solution (50 mL of phosphoric acid + 40 mL of glycerin, with a volume fixed to 1000 mL) and

placed inside a rigid plastic tube. The lower sponge was positioned 5 cm above the base of the tube, and the upper sponge was positioned flat against the top of the tube [44]. The samples were collected on the following day at 8:00. During sampling, the lower sponge was expeditiously transferred and segregated into a sanitized plastic pouch. In the meantime, another sponge soaked in phosphoglyceride replaced the previous one. The  $\text{NH}_4^+$ -N concentration in the sponge was analyzed by subjecting it to a 1 M KCl extraction using a mechanical shaker at 25 °C for 60 min, and then analyzing the filtrates using a continuous chemical analyzer (SmartChem200, Roma, Italy), while the  $^{15}\text{N}$  enrichment of extractable  $\text{NH}_4^+$ -N ascertained by the KCl extract was concentrated using a modified diffusion method described in [45]. Subsequently, the extract was analyzed using a Finnigan Delta-plus XP stable isotope-ratio mass spectrometer manufactured by Thermo Finnigan, Silicon Valley, CA, USA.

Flux and cumulation of  $\text{NH}_3$  volatilization derived from soil and labeled urea N were calculated using the following formula [46].

$$\text{Soil ammonia volatilization flux } (F_{\text{SNH}_3}, \text{ g N ha}^{-1} \text{ day}^{-1}) = \frac{M}{A \times D}$$

where  $M$  is the average ammonia amount ( $\text{NH}_3$ -N, mg) measured using a single device via the closed ventilation method;  $A$  is the cross-sectional area of the capture device ( $\text{m}^2$ ); and  $D$  is the duration of each successive capture.

$$\text{Cumulation of soil ammonia volatilization } (C_{\text{SNH}_3}, \text{ g N ha}^{-1}) = \sum F_{\text{SNH}_3} \times D$$

$$\text{Urea derived ammonia volatilization flux } (F_{15\text{NH}_3}, \text{ g N ha}^{-1} \text{ day}^{-1}) = F_{\text{SNH}_3} \times \frac{a - c}{b - c}$$

where  $a$  is the  $^{15}\text{N}$  abundance of extractable  $\text{NH}_4^+$ -N for each sampling (%);  $b$  represents the  $^{15}\text{N}$  abundance of urea applied in 2016 (9.80%); and  $c$  represents the natural  $^{15}\text{N}$  abundance (0.366%).

$$\text{Cumulation of urea derived ammonia volatilization } (C_{15\text{NH}_3}, \text{ g N ha}^{-1}) = \sum F_{15\text{NH}_3} \times D$$

$$\text{Percentage of urea derived ammonia volatilization to total soil ammonia volatilization } (\%) = \frac{C_{15\text{NH}_3}}{C_{\text{SNH}_3}}$$

#### 2.4. Statistical Analysis

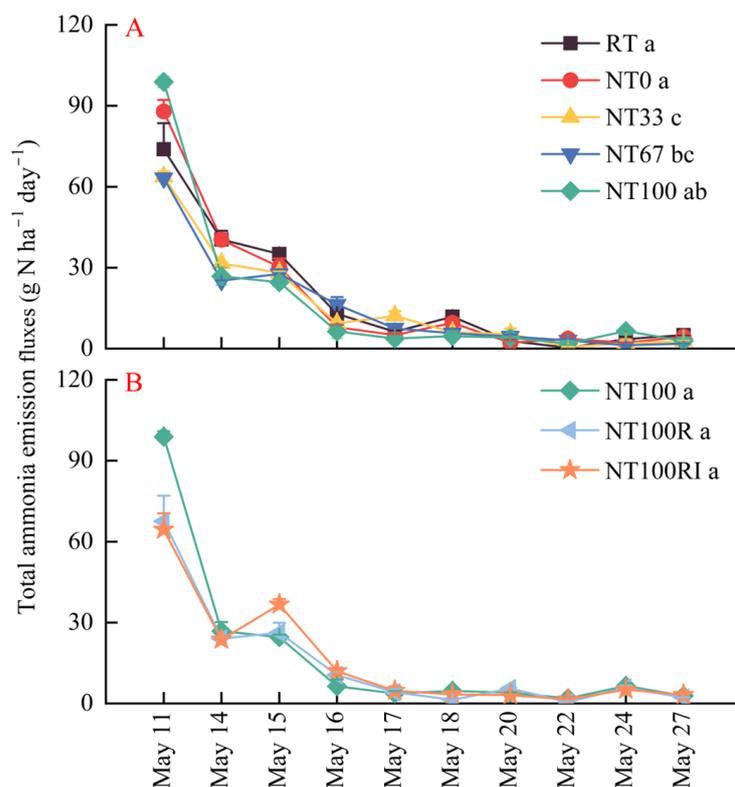
A completely randomized block design was adopted in this field study, so the treatments and blocks were treated as fixed and random factors, respectively, using the linear mixed-effects model. One-way analysis of variance (ANOVA) was used to evaluate the impacts of the treatments on cumulative  $\text{NH}_3$  and  $^{15}\text{N}$ -labeled  $\text{NH}_3$  emissions, as well as the ratios of  $^{15}\text{N}$ -labelled  $\text{NH}_3$  to  $\text{NH}_3$  emissions. The repeated-measures one-way ANOVA was used to evaluate the effects of the treatments on  $\text{NH}_3$  and  $^{15}\text{N}$ -labeled  $\text{NH}_3$  emission rates for 17 days of monitoring. One-way ANOVA with repeated measures was used to compare group means, where the participants were the same in each group, and means were measured multiple times to observe changes to an intervention. Differences were considered statistically significant at  $p < 0.05$ .

### 3. Results

#### 3.1. Total $\text{NH}_3$ and Fertilizer-Derived $^{15}\text{N}$ - $\text{NH}_3$ Emission Rates

Generally, the  $\text{NH}_3$  volatilization rates across all treatments were relatively high during the first five days after the application of urea, and gradually decreased from  $74.22 \text{ g N ha}^{-1} \text{ day}^{-1}$  on day 1 to  $10.77 \text{ g N ha}^{-1} \text{ day}^{-1}$  on day 6, and they tended to stabilize between  $1.65$  and  $6.21 \text{ g N ha}^{-1} \text{ day}^{-1}$  with the extension of time to day 17 (Figure 2). Specifically, the repeated-measures ANOVA showed that the  $\text{NH}_3$  emission rates throughout the 17-day

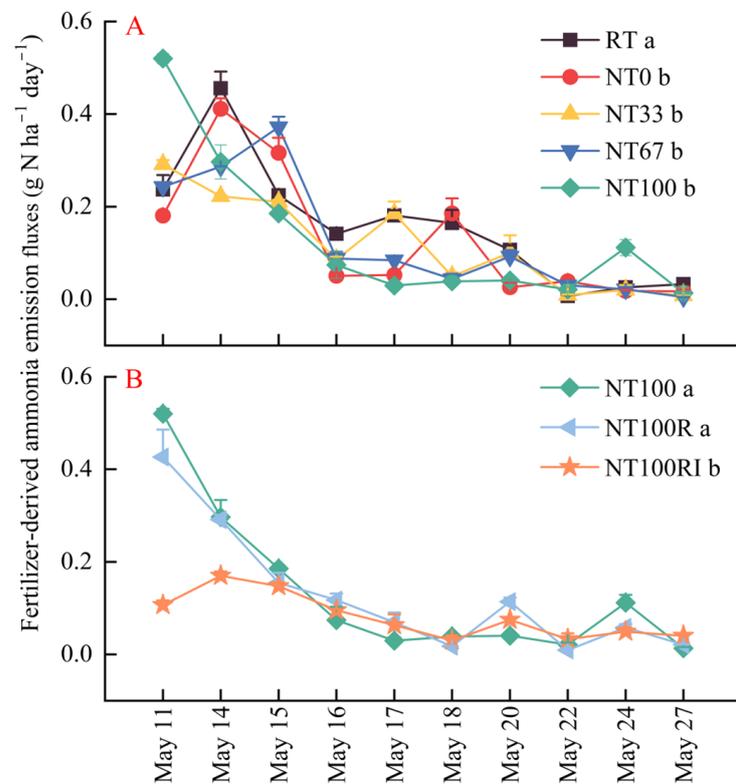
monitoring in conventional tillage of RT ( $19.22 \text{ g N ha}^{-1} \text{ day}^{-1}$ ) and no-till farming without mulching of NT0 ( $19.34 \text{ g N ha}^{-1} \text{ day}^{-1}$ ) were significantly greater than no-till farming with mulching of NT33 ( $16.18 \text{ g N ha}^{-1} \text{ day}^{-1}$ ) and NT67 ( $15.62 \text{ g N ha}^{-1} \text{ day}^{-1}$ ) (Figure 2A,  $p < 0.05$ ), although NT100 ( $18.04 \text{ g N ha}^{-1} \text{ day}^{-1}$ ), NT100R ( $14.81 \text{ g N ha}^{-1} \text{ day}^{-1}$ ), and NT100RI ( $15.77 \text{ g N ha}^{-1} \text{ day}^{-1}$ ) showed no significant difference between each other (Figure 2B).



**Figure 2.** Variation in total ammonia emission fluxes during the seeding stage of spring maize. The conventional and conservation tillage treatments were placed into group (A) (RT, NT0, NT33, NT67, and NT100), and conservation tillage combined with other measures were arranged into group (B) (NT100, NT100R, and NT100RI). Different lower case letters indicate significant differences at  $p < 0.05$ . Error bars indicate standard errors ( $n = 4$ ). RT: conventional ridge tillage; NT0: no-tillage farming with 0% maize straw mulching; NT33: no-tillage farming with 33% ( $2500 \text{ kg ha}^{-1}$ ) maize straw mulching; NT67: no-tillage farming with 67% ( $5000 \text{ kg ha}^{-1}$ ) maize straw mulching; NT100: no-tillage farming with 100% ( $7500 \text{ kg ha}^{-1}$ ) maize straw mulching; NT100R: NT100 plus 20% reduced fertilization; NT100RI: NT100R plus urease/nitrification inhibitors.

Similar to the  $\text{NH}_3$  volatilization rates, the fertilizer-derived  $^{15}\text{N-NH}_3$  emission rates across all treatments were higher for the first five days after the application of urea, although large fluctuation existed throughout the 17-day monitoring period (Figure 3). For ease of comparison, we grouped the conventional and conservation tillage treatments into one group (RT, NT0, NT33, NT67, and NT100) (Figure 3A), and the treatments where conservation tillage was combined with other measures were arranged into another group (NT100, NT100R, and NT100RI) (Figure 3B). Overall, the repeated-measures ANOVA indicated that no-tillage farming with all four levels of stover mulching, i.e., NT0, NT33, NT67, and NT100 ( $0.12\text{--}0.13 \text{ g N ha}^{-1} \text{ day}^{-1}$ ), could significantly reduce  $^{15}\text{N-NH}_3$  volatilization rates in comparison with the conventional tillage of RT ( $0.16 \text{ g N ha}^{-1} \text{ day}^{-1}$ ) (Figure 3A,  $p < 0.05$ ). In addition, the fertilization reduction and urease/nitrification inhibitors of NT100RI further reduced the  $^{15}\text{N-NH}_3$  volatilization rates (from  $0.13 \text{ g N ha}^{-1} \text{ day}^{-1}$  to

0.08 g N ha<sup>-1</sup> day<sup>-1</sup>) by 38%, compared with those associated with NT100 and NT100R (Figure 3B,  $p < 0.05$ ).

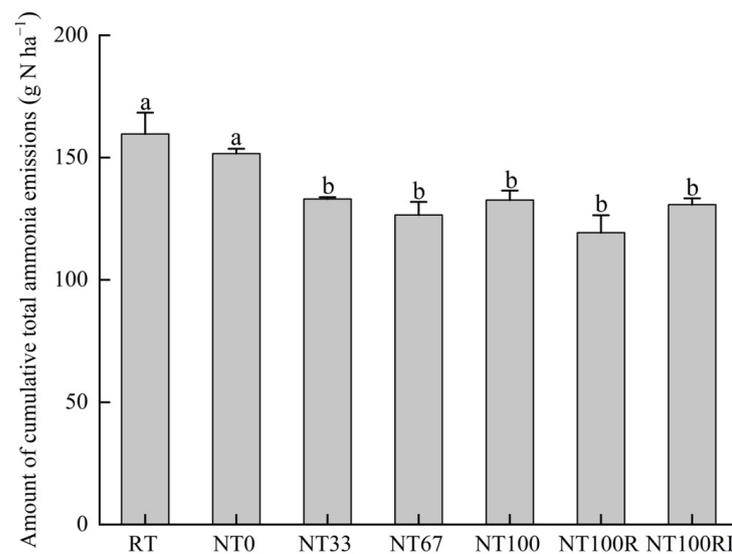


**Figure 3.** Variation in fertilizer-derived ammonia emission fluxes during the seeding stage of spring maize. The conventional and conservation tillage treatments were placed into group (A) (RT, NT0, NT33, NT67, and NT100), and conservation tillage combined with other measures were arranged into group (B) (NT100, NT100R, and NT100RI). Different lower case letters indicate significant differences at  $p < 0.05$ . Error bars indicate standard errors ( $n = 4$ ).

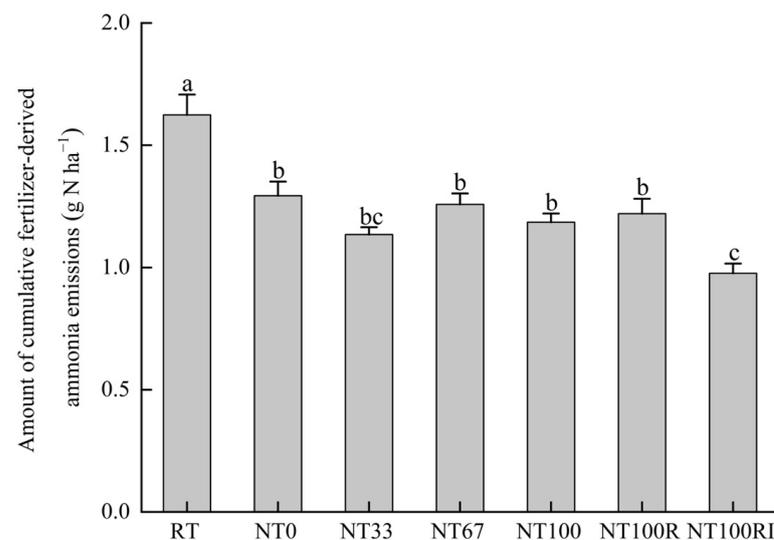
### 3.2. Cumulative Total NH<sub>3</sub> and Fertilizer-Derived <sup>15</sup>N-NH<sub>3</sub> Emissions

To better understand the NH<sub>3</sub> volatilization reduction, cumulative NH<sub>3</sub> emissions were calculated for each treatment of 17 days after the application of urea (Figure 4). Soil NH<sub>3</sub> volatilization was highest in the RT treatment (159.66 g N ha<sup>-1</sup>), followed by the NT0 treatment (151.62 g N ha<sup>-1</sup>). Compared to RT and NT0, the no-till and stover mulching treatments of NT33 (133.07 g N ha<sup>-1</sup>), NT67 (126.54 g N ha<sup>-1</sup>), and NT100 (132.66 g N ha<sup>-1</sup>) all significantly reduced cumulative NH<sub>3</sub> volatilization by averaging 16% ( $p < 0.05$ ). However, the NH<sub>3</sub> volatilization of NT100R (119.30 g N ha<sup>-1</sup>) and NT100RI (130.80 g N ha<sup>-1</sup>) was not significantly different from NT100. There was no significant difference between RT and NT0, nor between NT33, NT67, NT100, NT100R, and NT100RI.

To investigate the NH<sub>3</sub> volatilization loss directly from urea, cumulative <sup>15</sup>N-NH<sub>3</sub> emissions were calculated for each treatment of 17 days after the application of urea (Figure 5). The <sup>15</sup>N-NH<sub>3</sub> volatiles of RT were significantly higher than those of the other treatments, while there was no significant difference between NT0, NT33, NT67, NT100, and NT100R. Compared to the conventional tillage of RT (1.62 g N ha<sup>-1</sup>), no-tillage treatment of NT0 (1.29 g N ha<sup>-1</sup>) and no-till with mulching treatments of NT33 (1.13 g N ha<sup>-1</sup>), NT67 (1.26 g N ha<sup>-1</sup>), and NT100 (1.19 g N ha<sup>-1</sup>) all significantly reduced cumulative <sup>15</sup>N-NH<sub>3</sub> emissions by an average of 25% ( $p < 0.05$ ). More importantly, urease/nitrification inhibitors of NT100RI (0.98 g N ha<sup>-1</sup>) further reduced <sup>15</sup>N-NH<sub>3</sub> volatilization by 20% compared with the fertilization reduction treatment of NT100R (1.22 g N ha<sup>-1</sup>) ( $p < 0.05$ ).

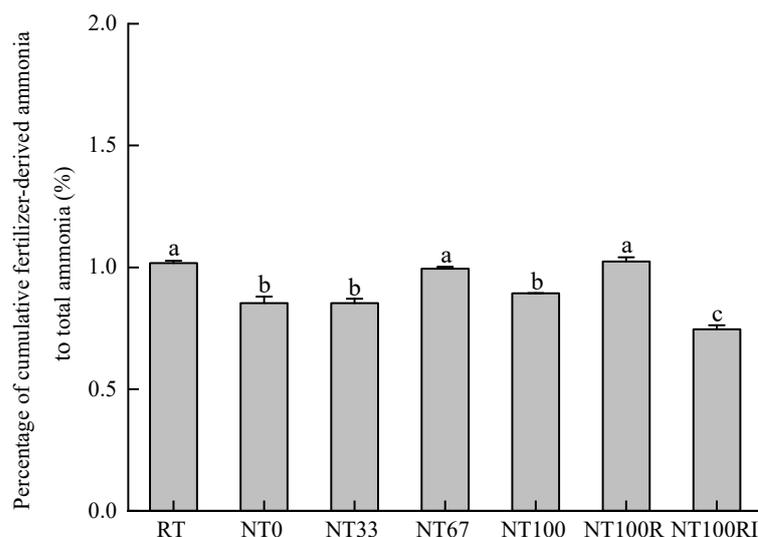


**Figure 4.** Amount of cumulative total ammonia emissions during the seeding stage of spring maize. Different lower case letters indicate significant differences at  $p < 0.05$ . Error bars indicate standard errors ( $n = 4$ ).



**Figure 5.** Amount of cumulative fertilizer-derived ammonia emissions during the seeding stage of spring maize. Different lower case letters indicate significant differences at  $p < 0.05$ . Error bars indicate standard errors ( $n = 4$ ).

To determine the extent of  $\text{NH}_3$  volatilization loss directly from urea, the ratios of  $^{15}\text{N-NH}_3$  to  $\text{NH}_3$  emissions were calculated for each treatment of the 17 days after the application of urea (Figure 6). Compared to the conventional tillage of RT (1.02%), no-tillage treatment of NT0 (0.85%) and no-till with mulching treatments of NT33 (0.85%) and NT100 (0.89%) all significantly reduced the ratios of  $^{15}\text{N-NH}_3$  to  $\text{NH}_3$  emissions by averaging 15% ( $p < 0.05$ ). Interestingly, the percentage was 15% greater in the reduced fertilization treatments of NT100R (1.02%), compared to the NT100 treatment, while reduced fertilization and urease/nitrification inhibitors of NT100RI (0.75%) reduced the percentages by 20%, compared with the fertilization reduction treatment of NT100R ( $p < 0.05$ ).



**Figure 6.** Percentage of cumulative fertilizer-derived ammonia with respect to total ammonia during the seeding stage of spring maize. Different lower case letters indicate significant differences at  $p < 0.05$ . Error bars indicate standard errors ( $n = 4$ ).

#### 4. Discussion

Fertilizers are easily lost through various ways, so reducing fertilizer loss caused by  $\text{NH}_3$  volatilization can improve fertilizer utilization rates and reduce subsequent air and water pollution. This study investigated whether a series of conservation management measures, used alone or in combination, would have an impact on  $\text{NH}_3$  volatilization.

The tillage technique, fertilization timing, mulching technique, and other factors may have an impact on the  $\text{NH}_3$  emission rates. In our study,  $\text{NH}_3$  emission rates in all treatments decreased over time. This could be because urea hydrolyzes quickly in soil, releasing most  $\text{NH}_3$  emissions in the first few days following application [41]. Consistent with our research, when urea was applied to crop surface, researchers found that the loss rate of  $\text{NH}_3$  reached its peak at 23 h after application with most  $\text{NH}_3$  loss occurring within 50 h [47]. Due to biological processes, such as nitrogen infiltration, nitrification, and denitrification as well as plant and animal absorption and consumption, the substrate's content reduces with time [48], so the rate of  $\text{NH}_3$  volatilization is highest in the days after urea application, and then gradually drops to a lower and stable level [49]. In conclusion, the volatilization of  $\text{NH}_3$  usually occurs within two weeks after the application of fertilizer in the studied Mollisols, so although our experimental period was relatively short, it was already possible to cover a clear process of  $\text{NH}_3$  volatilization and the final results were significant.

The results of earlier research on background  $\text{NH}_3$  emissions were varied. One study reported that the background  $\text{NH}_3$  emission levels of croplands in China reached  $0.99\text{--}2.18 \text{ Tg N yr}^{-1}$  [2]. However, other studies have suggested that  $\text{NH}_3$  volatilization is predominantly caused by the application of base fertilizer during crop planting [50], and that the background  $\text{NH}_3$  volatilization of unfertilized plots is often below the detection limit [29]. In our study, two weeks after fertilization, the total  $\text{NH}_3$  emissions ranged from  $126$  to  $160 \text{ g N ha}^{-1}$ , while the  $^{15}\text{N-NH}_3$  emissions derived from fertilizer ranged from  $0.98$  to  $1.62 \text{ g N ha}^{-1}$ . Although the degree of volatilization of  $\text{NH}_3$  in our study was very small, there was still a significant decreasing trend associated with six different experimental treatments. The low amount of  $\text{NH}_3$  volatilization observed may be related to the combined effects of conservation tillage, the reduced application of fertilizer, and the dual inhibitors employed. This low level of  $\text{NH}_3$  volatilization was also corroborated by an earlier study carried out at the same location as ours [51]. Therefore, it is possible to further limit the loss of N fertilizer through the hydrolysis of ammonia by combining other N management

methods, such as urease/nitrification inhibitors, reduced fertilization, conservation tillage, and other agricultural measures [52], which would improve fertilizer utilization.

In our study, in addition to no tillage farming alone, it was determined that treatment under conservation tillage can significantly reduce total  $\text{NH}_3$  and  $\text{NH}_3$  emissions from fertilizer, and the addition of dual inhibitors can further reduce  $\text{NH}_3$  emissions from fertilizer. This may be because maize straw with high C/N ratios, combined with N inputs from fertilizers, provides a sufficient C substrate from which soil microorganisms can assimilate native soil N, reducing nitrate leaching and gaseous N emission, and thereby reducing the potential for N loss [53–55]. However, previous studies showed that conservation tillage management is not always effective in reducing the volatilization of  $\text{NH}_3$ . For example, contrary to the results of our study, it was reported that no-till soils seeded with urea presented a loss of  $\text{NH}_3$  volatilization 1.3 times that of reduced-till soils [56];  $\text{NH}_3$  volatilization in no-till soil even accounted for 24% of topdressing urea [57], and sometimes the cumulative volatilization of  $\text{NH}_3$  can even reach three times higher than those in conventionally tilled soils [58]. The reason behind this discrepancy may be that no-till practices typically apply urea to the soil surface, while crop residues act as a contact barrier between urea and soil, resulting in reduced nitrogen permeability and soil adsorption of  $\text{NH}_4^+$  and the increased volatilization of  $\text{NH}_3$  [59]. However, in our study, fertilizer was applied at a soil depth of 10–15 cm, which might have reduced the exposure of the fertilizer to the air and thus reduced  $\text{NH}_3$  volatilization. Previous studies also showed that when urea is deeply buried in the soil, the top-cover materials of maize straw can act as a physical barrier that significantly reduces N loss [60,61]. Our previous studies showed that stover mulching can regulate the active organic N from soil mineral N pools to more stable soil-fixed  $\text{NH}_4^+$  and organic N pools, in which N can be slowly released or mineralized into plant available pools, resulting in higher fertilizer N-use efficiency in soil-crop systems [42], and thereby reducing the volatilization of  $\text{NH}_3$ . Furthermore, combining no tillage farming with stover mulching can regulate soil microbial growth and crop stover decomposition [20,62], affecting the urea hydrolysis process that controls  $\text{NH}_3$  volatilization from the applied urea.

To accurately quantify  $\text{NH}_3$  emissions stemming from fertilizer,  $^{15}\text{N}$ -labeled tracer techniques must be used to measure gaseous N loss in the form of  $\text{NH}_3$  directly derived from fertilizers [10]. Our results showed that although the variation in the volatilization rate of  $^{15}\text{N}$ - $\text{NH}_3$  after fertilization was less obvious than that of the overall  $\text{NH}_3$  emission rate; the volatilization rate still showed a trend of reduction and stabilization with the extension of time (Figure 3). Compared to the other six treatments, the emission rate of  $^{15}\text{N}$ - $\text{NH}_3$  was significantly reduced on the first day after fertilization for NT100RI, and the overall change in the  $^{15}\text{N}$ - $\text{NH}_3$  emission rate was stable. This indicates that the dual inhibitor effectively inhibited the emission of  $\text{NH}_3$ . Previous studies also demonstrated that dual inhibitors delay  $\text{NH}_3$  volatilization and reduce the  $\text{NH}_3$  volatilization peak, and one application of a urease inhibitor is generally regarded to be a more effective way of reducing  $\text{NH}_3$  volatilization [41,49]. However, because no single urease inhibitor or nitrification inhibitor was used in this experiment, the effect of a single inhibitor combined with other agricultural management strategies on  $\text{NH}_3$  volatilization must be examined further. Previous research has demonstrated that  $\text{NH}_3$  emissions increase exponentially in response to higher nitrogen inputs [63]. Therefore, the volatilization of  $\text{NH}_3$  is largely dependent on the quantity of nitrogen fertilizer applied [64]. However, compared to the conservation tillage treatments alone, the combination of conservation tillage methods with a 20% reduction in fertilization had no discernible impact on total  $\text{NH}_3$  and  $^{15}\text{N}$ - $\text{NH}_3$  volatilization in our study (Figures 4 and 5). This may be because a 20% reduction in nitrogen inputs may not be sufficient to significantly reduce  $\text{NH}_3$  volatilization. Moreover, the amount of nitrogen lost through  $\text{NH}_3$  volatilization of surface-applied nitrogen fertilizer can also be influenced by a range of complex soil and environmental factors in the field [65]. Interestingly, the application of dual inhibitors significantly reduced  $^{15}\text{N}$ - $\text{NH}_3$  emissions and the percentage of  $^{15}\text{N}$ - $\text{NH}_3$  to  $\text{NH}_3$  emissions, compared with NT100 and NT100R under conditions

of reduced fertilizer application and conservation tillage practices (Figures 5 and 6). It was further demonstrated that the dual inhibitor effectively reduced  $\text{NH}_3$  volatilization from the fertilizer. This result might have occurred because the dual inhibitor was buried deep underground with urea and directly affected the loss of  $\text{NH}_3$  in the fertilizer [66]. Some studies also showed that a combination of urease/nitrification inhibitors can reduce  $\text{NH}_3$  emissions [67,68]; however, it was also reported that the combination of urease and nitrification inhibitors may prolong the retention time of  $\text{NH}_4^+$  [69], thereby increasing  $\text{NH}_3$  emissions [30]. Our research supports the former because the heterogeneity of the environment can also potentially lead to different inhibitors having different effects on  $\text{NH}_3$  emissions [70]. For example, in Zaman's outdoor studies [40], rainfall occurred after fertilization, preventing the fertilizer from fully entering the soil and contributing to the rise in  $\text{NH}_3$  volatilization following the application of dual inhibitors. Moreover, previous studies showed that, when compared with the urease inhibitor alone, the simultaneous application of urease and nitrification inhibitors can promote  $\text{NH}_3$  volatilization, but also inhibit the volatilization of  $\text{NH}_3$  compared to controls without either inhibitor [41].

## 5. Conclusions

Our long-term conservation tillage study shows that the time of greatest  $\text{NH}_3$  volatilization was concentrated in the first five days after N fertilization. Using  $^{15}\text{N}$ -labeled tracer techniques, we found that conservation tillage can reduce the total  $\text{NH}_3$  and  $\text{NH}_3$  volatilization from fertilizers. On the basis of conservation tillage, the loss of  $\text{NH}_3$  volatilization in fertilizers can be further reduced through the application of urease/nitrification inhibitors. This detailed assessment of  $\text{NH}_3$  volatilization could be helpful in a range of issues regarding the loss of fertilizer in soil as  $\text{NH}_3$ . Our findings can provide a scientific basis for understanding gaseous N loss under a conservation management strategy, consisting of no-tillage farming and crop residue application with implications for the development of sustainable agriculture.

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