

INFLUENCE OF N SPLIT APPLICATION ON NH₃ VOLATILIZATION LOSSES AND N RECOVERY EFFICIENCY FROM PLASTIC MULCHING MAIZE IN LOESS PLATEAU, CHINA

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Abstract. Ammonia (NH₃) volatilization is one of the main reasons of applied-N loss from agricultural cropping systems. Adjusting the application timing of N fertilizer to improve temporal synchronicity between crop-N demand and soil-N availability could result in higher nitrogen (N) recovery efficiency (NRE) and lower N losses. A two-year experiment was conducted to investigate the effect of split fertilizer N application on NH₃ volatilization, grain yield, and NRE. Four treatments were included according to the N application ratio: (i) a single application of total N fertilizer at sowing (N1), (ii) N applied with two splits at a ratio of 4:6 (N2), (iii) N applied with three splits at a ratio of 4:3:3 (N3), and (iv) no N applied as a control (N0). The results show that NH₃ fluxes peaked at 3-5 days after fertilization and then dropped sharply within the next week. NH₃ fluxes increased significantly with N application rate and were significantly and positively correlated with soil NH₄⁺-N content. Slight precipitation (< 10 mm) promoted NH₃ volatilization, while heavy precipitation (> 10 mm) restrained it. Applying N with two or three splits significantly increased the grain yield, total N uptake, and NRE of maize and significantly reduced NH₃ volatilization losses.

Keywords: *Dräger-tube method, N application time, N application rate, N loss, grain yield*

Introduction

Ammonia (NH₃) volatilization is a major pathway of nitrogen (N) losses in agricultural systems worldwide, which results in low fertilizer N recovery efficiency (NRE) and many other environmental issues, such as soil acidification and surface water eutrophication through N deposition (Behera et al., 2013). In addition, NH₃ is considered a dominant air pollution source, accounting for 25-60% of the total PM_{2.5} mass (Sutton et al., 2013), and could become a secondary source of N₂O (Sutton et al., 2008). Agricultural activities reportedly contribute up to 90% of the total NH₃ emissions to the atmosphere (Boyer et al., 2002), with the majority originating from livestock production and approximately 12% resulting from N fertilizer application (Ferm, 1998). An average of 18% of applied synthetic fertilizer N is estimated to be lost as NH₃ globally (Pan et al., 2016). China is the world's largest NH₃ emitter with annual emissions that are 2.7 and 3.0 times higher than those of the European Union and the

United States, respectively (Paulot et al., 2014). The total NH₃ emission of China was estimated to be 3.55 Tg N in 2005, with an estimated 13.2% of N from fertilizer NH₃ volatilizing (Zhang et al., 2011). The factors influencing NH₃ volatilization include environmental parameters, such as soil pH, moisture, and temperature as well as precipitation (Ma et al., 2010; Yang et al., 2015; Pan et al., 2016) and N fertilizer management practices, such as the use of different types of N fertilizer, N rate and application methods (Liu et al., 2015; Yang et al., 2019).

Urea is one of the most widely used N fertilizers worldwide because of its high N concentration, and its hydrolysis is known as a key process in inducing NH₃ volatilization by producing highly concentrated NH₄⁺-N with sharply increased pH (Black et al., 1987). NH₃ volatilization from urea accounts for approximately 10-35% of the N applied in field crop planting (Soares et al., 2012). Due to the different demand of plants for N at different growth stages, surplus nitrogen is a great environmental risk in that it can leach into groundwater or be lost to the atmosphere through NH₃ volatilization and denitrification. Thus, adjusting the application timing of N fertilizer and improving temporal synchronicity between crop-N demand and soil-N availability are key strategies for improving the NRE (Ribaud et al., 2011). Field experimental data have shown that grain yields, plant N uptake, and NRE increase with increases in topdressed N fertilizer (López-Bellido et al., 2005; Cui et al., 2008). Scharf et al. (2002) found that crop yields were still responsive to N application until the silking (R1) stage but that the potential yields were not achieved when N applications were delayed until that stage. Applying N with three splits could produce higher grain yields and higher cumulative N₂O emissions than a single application of N (Wang et al., 2016). Previous studies have shown that high rates of N lead to much higher NH₃ losses, and the emissions response to increasing N input is exponential rather than linear (Ma et al., 2010). Therefore, we hypothesize that maintaining a low rate of N application by using a split application should reduce NH₃ volatilization losses.

Plastic mulching is used worldwide in vegetable and grain production, especially in arid, semiarid and subhumid areas (Chen et al., 2014), due to its ability to increase soil temperature and moisture, reduce water evaporation, and improve soil nutrient availability (Li et al., 2004). In addition, as a physical barrier, plastic mulching restricts gas exchange between the soil and atmosphere, thus affecting NH₃ volatilization. However, there is little information concerning NH₃ volatilization losses from maize under plastic mulching. The objective of this study was to quantify the effect of N split application on NH₃ volatilization, crop yields, and NRE in a plastic mulching maize cropping system on the Loess Plateau of China.

Materials and methods

Site description

The field experiment was conducted at Changwu Agricultural and Ecological Experimental Station of Chinese Academy of Sciences (35.28°N, 107.88°E, 1200 m altitude) in 2014 and 2015, which has a semiarid climate on the Loess Plateau of China. From 2009 to 2013, the mean annual air temperature is 9.7 °C, and the average annual precipitation is 555 mm, 73% of this falls during the maize growth season (MS), whereas the average potential evaporation is 1560 mm. Total precipitation during MS was 375 mm in 2014, and 361 mm in 2015, respectively. The main cropping system in this area includes harvesting one crop of maize or wheat per year. The soil at the

experiment site is classified as Cumuli-Ustic Isohumosols according to the Chinese Soil Taxonomy (Gong et al., 2007). The soil properties at the top 20 cm were: bulk density 1.3 g cm⁻³, pH 8.3, organic C 8.1 g kg⁻¹, total N 1.0 g kg⁻¹, available phosphorus (Olsen-P) 21.5 mg kg⁻¹, and available potassium (NH₄OAc-K) 135.2 mg kg⁻¹, and mineral N (NO₃⁻-N + NH₄⁺-N) 28.3 mg kg⁻¹.

Experimental design

The experimental design consisted of a completely randomized block with three replicates and an area of 5 m × 6 m for each plot. The split application of N at the same rate of 225 kg ha⁻¹ was the major factor that was investigated. The four treatments were (i) no N as a control (N0), (ii) 100% N applied at sowing (N1), (iii) N applied at the sowing and jointing (V8) stages in a ratio of 4:6 (N2), and (iv) N applied at the sowing, V8 and R1 stages in a ratio of 4:3:3 (N3). Half-film mulching was performed for all of the plots; the width of the plastic film and the interval of two films were both 0.5 m, and maize was seeded on both sides of the film, with a row spacing of 0.5 m. For basal N, N fertilizer in the form of urea (N 46%) was manually distributed over the soil surface prior to sowing and then mixed with the soil of the 0-15 cm layer; for topdressed N, N fertilizer was applied in a band in the middle of the no-film rows at a depth of 5 cm. Each plot was supplied with 40 kg P ha⁻¹ (calcium superphosphate, P₂O₅, 12%) and 80 kg K ha⁻¹ (potassium sulfate, K₂O, 45%) at sowing with the base N fertilizer. A high-yielding maize hybrid (Pioneer 335) was used in this study; the plant density was 65,000 plants ha⁻¹.

NH₃ volatilization measurements

The calibrated Dräger-Tube method (DTM) was used to quantify NH₃ volatilization (Pacholski et al., 2006) as this method has been shown to be well suited for NH₃ measurements in multiplot field trials (Ni et al., 2014; Wolf et al., 2014). During the measurement, four stainless steel rings were installed in the upper soil of one plot for each treatment directly after fertilization, 2 on the edge of the mulch, and 2 on the bare land, then they were connected to an NH₃ indicator tube and an automatic pump to ensure a defined flow rate. Air was simultaneously sucked through four connected chambers and led through a NH₃ tube (*Fig. 1*). Damaged tubes were exchanged by new tubing. Using Dräger tubes for different NH₃ concentration ranges allowed measurements of NH₃ concentrations ranging from 0.1 to 70 ppm. In conjunction with all NH₃ measurements, wind speeds at heights of 2.0 m and 0.2 m were recorded every 10 min for the duration of every measuring period (approximately 10-14 days after N fertilizer application until NH₃ volatilization losses were no longer detected), which were used for calculating the calibrated NH₃ fluxes.

Soil property measurements

Soil samples at a 20 cm depth were taken from each plot for the moisture and NH₄⁺-N determinations, while NH₃ volatilization was measured using a 4-cm-diameter gauge auger. Each sample was a composite of two subsamples, of which one was taken from the no-mulch bands and the other was taken from film mulched bands, to represent the aggregate condition. The samples were oven-dried at 105 °C for 24 h to a consistent weight to determine the gravimetric soil water content, and the soil water-filled pore space (WFPS) was subsequently calculated using *Equation 1*. To determine the soil

NH₄⁺-N content, representative fresh subsamples (5 g) were extracted using 50 ml of a 1 mol L⁻¹ KCl solution, and the extracts were analyzed using an automated flow injection analyzer (FLOWSYS, Italy). The soil temperatures at the surface and at a depth of 10 cm were measured using portable digital thermometers (JM624, Jinming Instrument Ltd., Tianjin, China). Precipitation data were obtained from an automatic weather station near the experimental site.

$$WFPS = \frac{\text{Soil water content}(\%) \times \text{Soil bulk density}}{1 - \frac{\text{Soil bulk density}}{2.65}} \times 100\% \quad (\text{Eq.1})$$

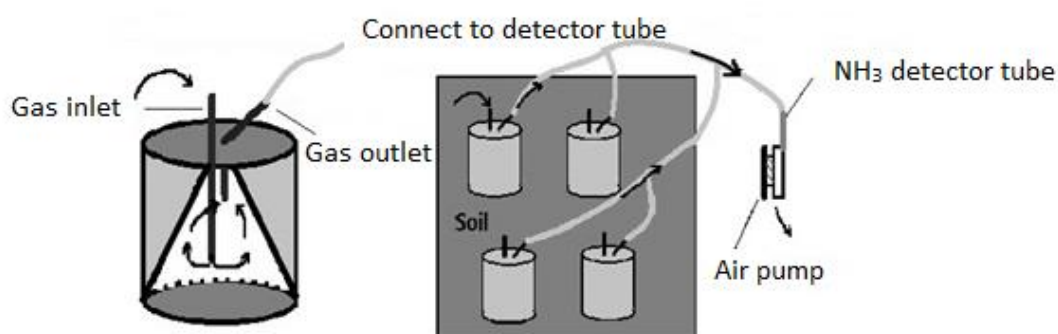


Figure 1. Schematic diagram of NH₃ volatilization measurements

Grain yield and N recovery efficiency

At harvest, 8 m² (4 rows each 4 m long) in the middle of each plot was manually harvested to determine the grain yield, and the grain yield was expressed at 15.5% moisture.

N recovery efficiency (NRE) was calculated by difference method using *Equation 2*.

$$NRE (\%) = \frac{N \text{ uptake in fertilized plot (kg hm}^{-2}\text{)} - N \text{ uptake in unfertilized plot (kg hm}^{-2}\text{)}}{N \text{ application rates (kg hm}^{-2}\text{)}} \quad (\text{Eq.2})$$

Statistical analysis

Statistical analysis was conducted using the SPSS 20.0 software package for one-way analysis of variance (ANOVA); the statistically significant differences between different treatments were tested by the least significant difference (LSD) at the 5% level. The Pearson correlation analysis was performed to investigate the correlations between the NH₃ volatilization flux and soil variables.

Results

Soil WFPS, temperature and NH₄⁺-N content

The soil WFPS and temperature during NH₃ volatilization measurements over two years are shown in *Figure 2*. The mean soil temperature during the application of basal N, topdressed N at the V8 stage (T_{V8-N}) and topdressed N at the R1 stage (T_{R1-N}) were 15.5 °C, 21.3 °C and 21.7 °C, respectively, in 2014 and were 15.6 °C, 18.3 °C and 18.4 °C, respectively, in 2015. Soil WFPS fluctuated with precipitation events, varying

from 20.6 to 59.4% in 2014 and from 36.7 to 64.4% in 2015, and there was no difference among the four treatments.

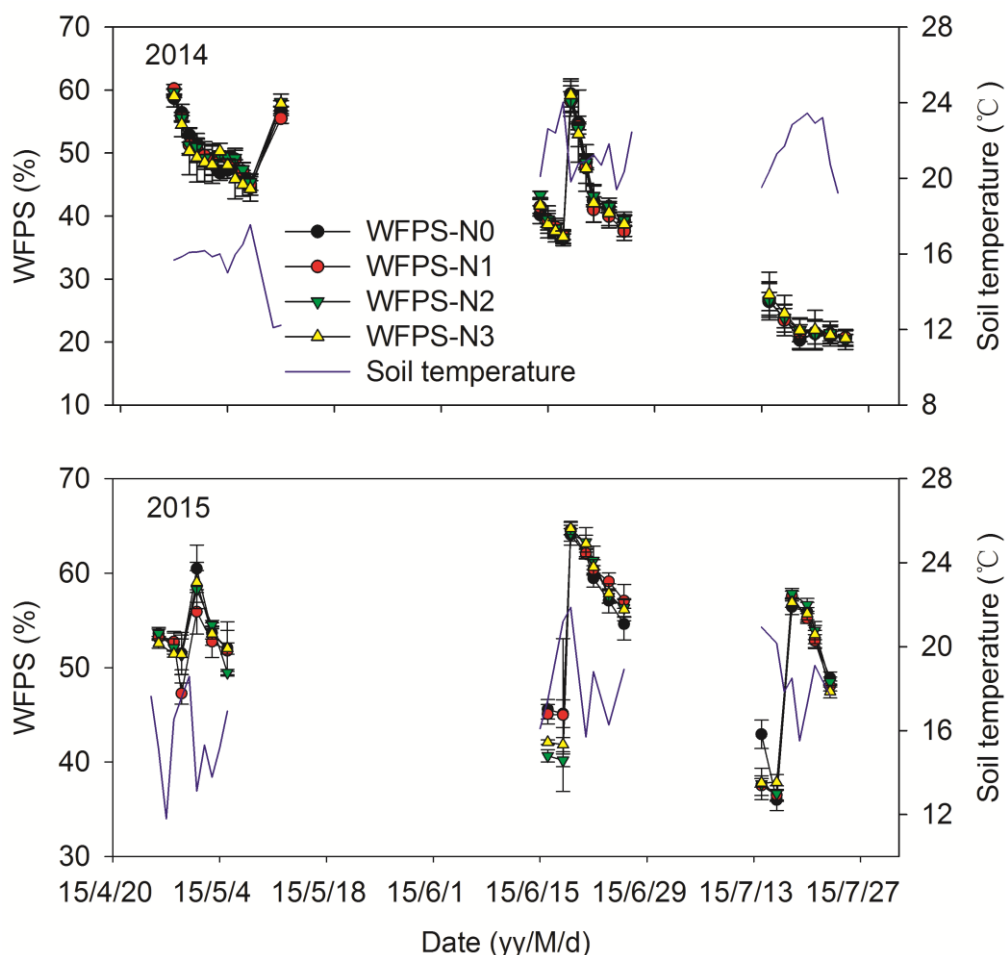


Figure 2. Soil moisture and temperature dynamics during NH_3 volatilization measurements in 2014 and 2015

The soil NH_4^+ -N content increased sharply after the application of N fertilizer and then returned to normal within approximately one week (*Fig. 3*). The mean soil NH_4^+ -N content during the basal N application in the N1 treatment was 66.1 mg kg^{-1} , which was significantly higher than that in the N2 (20.7 mg kg^{-1}) and N3 (17.9 mg kg^{-1}) treatments in 2014. Relatively low soil NH_4^+ -N content was measured during basal N application in 2015, with mean values of 21.6 , 5.1 and 4.2 mg kg^{-1} in the N1, N2 and N3 treatments, respectively. After the $\text{T}_{\text{V8-N}}$ application, the mean soil NH_4^+ -N content was 55.9 mg kg^{-1} and 26.1 mg kg^{-1} in treatments N2 and N3, respectively, in 2014 and 163.1 mg kg^{-1} and 77.7 mg kg^{-1} , respectively, in 2015, respectively. After the $\text{T}_{\text{R1-N}}$ application in the N3 treatment, the mean soil NH_4^+ -N content reached 33.2 mg kg^{-1} and 59.1 mg kg^{-1} in 2014 and 2015, respectively. Overall, the mean soil NH_4^+ -N contents across the whole growth period were 4.2 , 28.5 , 30.3 and 24.6 mg kg^{-1} in the N0, N1, N2 and N3 treatments, respectively, in 2014 and were 1.5 , 7.4 , 59.1 and 49.1 mg kg^{-1} , respectively, in 2015.

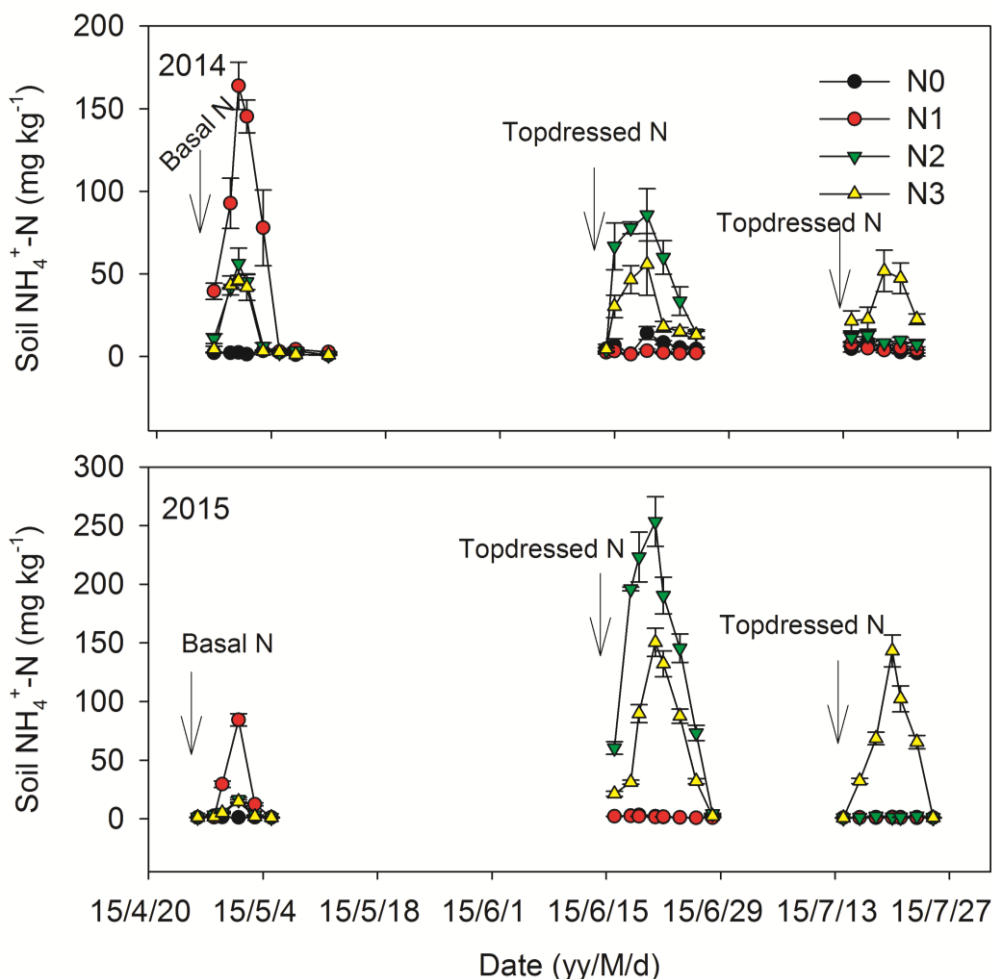


Figure 3. Soil NH₄⁺-N dynamics after the application of basal and topdressed N in 2014 and 2015. Bars denote standard deviations of soil NH₄⁺-N content on three replicate plots

NH₃ volatilization flux

The dynamics of NH₃ volatilization fluxes with different N split applications are shown in *Figure 4*. Since NH₃ volatilization mainly occurred after fertilization, it was only determined within two weeks after fertilization in this study. Under different N application periods, the rate of NH₃ volatilization usually peaked at 3-5 days after fertilization and then dropped sharply within the next week. After the basal N application, the mean NH₃ volatilization rate in the two years was 71 g ha⁻¹ h⁻¹ in the N1 treatment, which was significantly higher than the 5, 24, 27 g ha⁻¹ h⁻¹ in the N0, N2 and N3 treatments, respectively, due to its higher N application rate. The highest volatilization rates from N1 were 193 and 234 g ha⁻¹ h⁻¹ after the basal N applications in 2014 and 2015, respectively, while only 100-130 g ha⁻¹ h⁻¹ were observed in the N2 and N3 treatments. After the T_{V8}-N application, the mean rates of NH₃ volatilization from the N2 treatment were 18.8 and 26.5 g ha⁻¹ h⁻¹ in 2014 and 2015, respectively, which were significantly higher than those from the N3 treatment (10.4 g ha⁻¹ h⁻¹ in 2014 and 11.8 g ha⁻¹ h⁻¹ in 2015). A small emissions peak emerged in the N3 treatment when T_{R1}-N was applied, with the highest volatilization rates of 37.5 g ha⁻¹ h⁻¹ in 2014 and 77.5 g ha⁻¹ h⁻¹ in 2015.

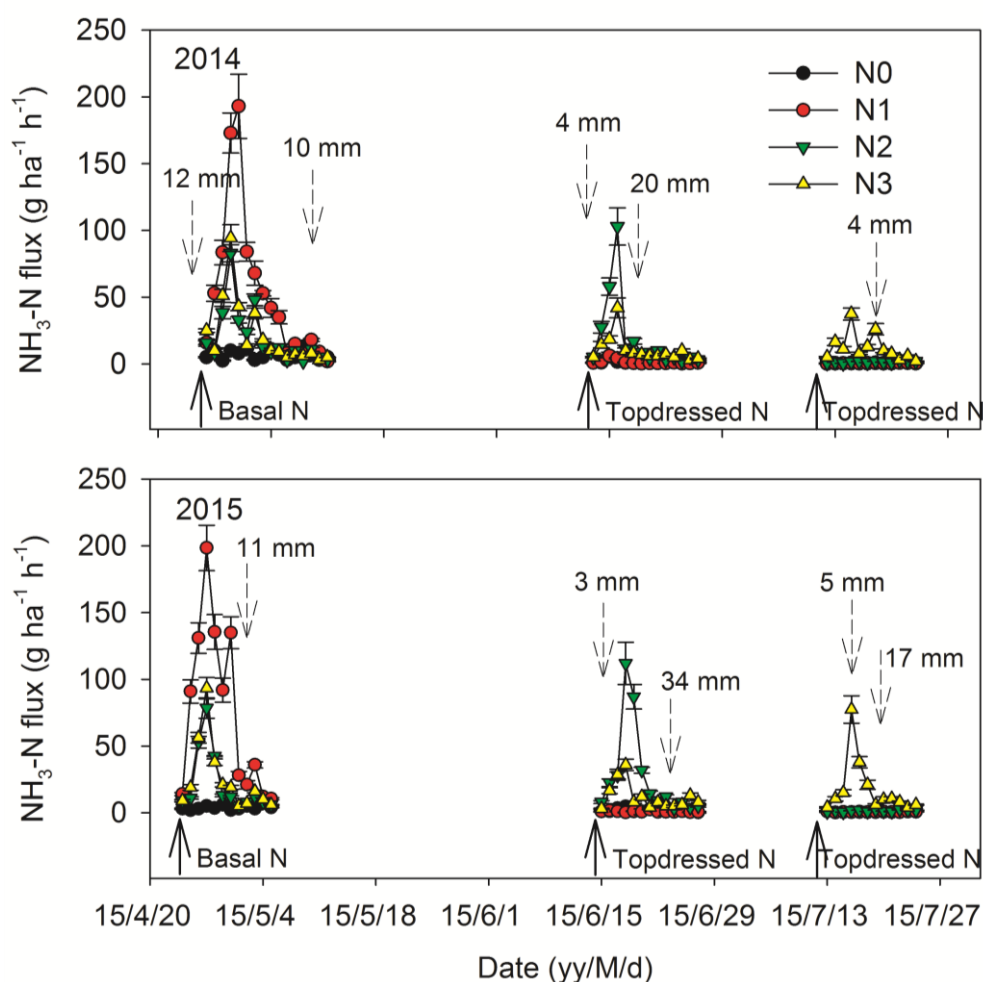


Figure 4. NH₃ volatilization fluxes after the application of basal and topdressed N in 2014 and 2015. Bars denote standard deviations of NH₃ fluxes on three replicate plots

The total NH₃ volatilization and fertilizer-induced emission (FIE) are shown in Table 1. The total NH₃ volatilizations under different N split applications were 13.5-20.4 kg ha⁻¹ and 14.2-21.5 kg ha⁻¹ in 2014 and 2015, respectively, accounting for 6.0%-9.1% and 6.3%-9.6% of the total N application rates, respectively. The amount of NH₃ volatilization increased with increasing N levels applied after the basal or topdressed N applications. Compared to the single N application (N1), N applications with two or three splits significantly reduced the NH₃ volatilization losses by 30.5%. The FIE of NH₃ from basal N was 7.6% (averaged over two years), which was significantly higher than that from topdressed N (5.2%) in the N2 treatment. For the N3 treatment, a higher FIE of NH₃ was measured from basal N (8.4%) than from topdressed N (5.7%).

The NH₃ volatilization flux was significantly and positively correlated with the soil NH₄⁺-N content, while no significant correlation was observed among the NH₃ volatilization flux, soil WFPS and temperature (Table 2).

Grain yield and N recovery efficiency

Compared with the N0 treatment, the N fertilizer application treatments significantly increased the grain yield of maize, varying from 12.4 to 13.3 t ha⁻¹ under different N

split applications over two years. The mean yields in the two years were 13.2 and 13.4 t ha⁻¹ in the N2 and N3 treatments, respectively, which were significantly higher than that of in the treatment N1 of 12.4 t ha⁻¹. The total N uptake varied from 193.9 to 238.5 kg ha⁻¹ in the fertilized treatments, and split N application significantly increased the total N uptake. The average NRE across two years was 47.3%, 58.8% and 67.1% in the N1, N2 and N3 treatments, respectively. In comparison to N1, applying N with two or three splits increased the NRE significantly.

Table 1. Accumulated NH₃ volatilization and fertilizer induced emission (FIE) from basal and topdressed N fertilizer

Treatments		N rate (kg ha ⁻¹)	Total NH ₃ volatilization (kg ha ⁻¹)		Fertilizer-induced emission (%)	
			2014	2015	2014	2015
N1		225	20.4 aA	21.5 aA	9.1 aA	9.6 aA
N2	B	90	7.1 b	6.5 b	7.9 a	7.2 b
	T _{V8}	135	6.4 b	7.7 b	4.7 b	5.7 c
	Total	225	13.5 B	14.2 B	6.0 B	6.3 B
N3	B	90	7.8 b	7.3 b	8.6 a	8.1 b
	T _{V8}	67.5	3.4 c	3.5 c	5.0 b	5.2 c
	T _{R1}	67.5	3.6 c	4.9 c	5.4 b	7.3 b
	Total	225	14.8 B	15.7 B	6.6 B	7.0 B

Different lowercase letters within a column indicate significant differences ($p < 0.05$). Different capital letters within a column indicate significant differences ($p < 0.05$). B denotes basal N; T_{V8} and T_{R1} denote topdressed N at the eight-leaf stage and the silking stage, respectively

Table 2. The relationship between NH₃ volatilization flux and soil properties

	Soil NH ₄ ⁺ -N	WFPS	Soil temperature
NH ₃ flux	0.288**	0.021	-0.089

**Correlation is significant at the 0.01 level

Discussion

Urea applied to soil will be rapidly hydrolyzed into ammonium nitrogen at appropriate temperature and humidity and then easily converted into NH₃ volatilization loss (Li et al., 2018). The total cumulative NH₃ loss under different N split applications ranged from 6.2 to 9.4% of the applied N in this study (Table 1), which were slightly lower than the emissions factor of 10% of applied N advocated by the IPCC (2007). A lower FIE of less than 2.5% has also been reported by another study on the Loess Plateau in rain-fed winter wheat field (Yang et al., 2015). Cai et al. (2002) found that the NH₃ volatilization losses range from 11 to 48% of the applied N to maize. The lower NH₃ losses in the present study than in Cai et al. (2002) may be attributed to the application method of N fertilizer. In this study, basal N was broadcast on the surface and then was mixed into soil; for topdressed N, N fertilizer was applied in a band in the middle of the no-film rows at a depth of 5 cm. NH₃ volatilization losses of N fertilizer are closely related to the application technique and N rate (Sommer et al., 2003; Zhao et al., 2009). In comparison to surface application, deep placement of N fertilizer could

reduce NH₃ losses substantially (Zeng et al., 2016). Rochette et al. (2013) reported that NH₃ volatilization losses were 50% of applied N when urea was banded at the surface and incorporation of the band decreased emissions by an average of 7% cm⁻¹. Pan et al. (2016) also found that in comparison to surface application, deep placement significantly decreased NH₃ volatilization through the incorporation of fertilizer at 54.7%.

A previous study reported that a split applications of N fertilizer did not affect NH₃ volatilization, regardless of splitting frequency (Pan et al., 2016). In our study, applying N with two or three splits significantly decreased NH₃ volatilization by 30.5% over two years, and the FIE from topdressed N was significantly lower than that from basal N (*Table 1*). One possible reason for this discrepancy is the different application method with basal and topdressed N, as described above. Basal N was broadcast on the surface and then mixed with the 0-15 cm soil; however, some of the N fertilizer was still exposed on the soil surface, increasing the risk of NH₃ losses compared to that with topdressed N. In addition, N losses through NH₃ volatilization increase with an increasing rate of N application in soils (Tian et al., 2001; Zhao et al., 2009), and previous studies have shown that the NH₃ emissions response to increasing N input is exponential rather than linear (Ma et al., 2010). In the present study, all N fertilizer was applied as basal dressing in treatment N1, which led to large amounts of NH₃ volatilization.

NH₃ volatilization is a physical process that is mainly influenced by the concentration of NH₄⁺-N in the soil solution and by the resistance to NH₃ movement from the soil matrix (Sommer et al., 2004). Rochette et al. (2013) found cumulative losses increased exponentially with increasing maximum NH₄⁺-N measured in the surface soil during the experiment. Consistent with previous reports, significant and positive correlations were observed between NH₃ volatilization flux and soil NH₄⁺-N contents in this study (*Table 2*). Soil temperature and moisture are two other important factors affecting NH₃ volatilization. Fan et al. (2011) reported that the cumulative NH₃ volatilization loss increased by 1.6 fold in sandy soil when the temperature increased from 20 to 30 °C. A study in the wheat growing season showed that soil temperature has positive effects on NH₃ fluxes during Oct-Dec but has negative effects during Feb.-May (Yang et al., 2015). However, no significant correlations were observed between NH₃ flux and soil temperature in the present study. This difference could be explained by the influence of precipitation events on the soil temperature and NH₃ volatilization flux. In some cases, high NH₃ emissions occurred following precipitation events when the soil temperature was relatively low. Soil moisture had significant positive effects on NH₃ fluxes, which could explain 7.3-19.7% of the variation in the NH₃ fluxes (Yang et al., 2015). Bosch-Serra et al. (2014) found that NH₃ volatilization was significantly reduced when the WFPS in the 0-30 cm soil layer was less than 56%. In this study, no significant correlations were observed between the NH₃ flux and soil WFPS. This result may be attributed to the different effects of precipitation amount on NH₃ fluxes. Previous studies have reported that the magnitude of NH₃ losses was largely influenced by small temporal differences in the weather and initial soil moisture content (Engel et al., 2011; Turner et al., 2012). The largest losses (30-44% of applied N) occurred after urea was applied to high-water content soil surfaces, followed by a period of slow drying with little or no precipitation (Engel et al., 2011). Light precipitation and high soil moisture would increase NH₃ volatilization risk by promoting urea hydrolysis, while heavy precipitation may effectively mitigate NH₃ losses by leaching

unhydrolyzed urea into the soil profile (Holcomb et al., 2011). In this study, the NH₃ flux increased sharply following slight precipitation events of 3 mm and 5 mm after topdressed N was applied at the V8 and R1 stages, respectively, in 2015. We also observed that the NH₃ flux was restrained rapidly by heavy precipitation of 20 mm after the application of topdressed N at the V8 stage in 2014 (Fig. 4).

Adjusting the application timing of N fertilizer and improving temporal synchronicity between crop-N demand and soil-N availability is a key strategy for improving the NRE (Ribaud et al., 2011). In the present study, higher grain yields, total N uptake, and NRE were observed from the plots under N split applications than in the other plots (Table 3). Similar to our results, Shi et al. (2012) also found that the grain yield and NRE increased significantly when N fertilizer application was divided into an appropriate ratio of basal and topdressed N. Yi et al. (2008) reported that the NRE increased by more than 2 fold when N was applied with splits of 1:1 compared with a split of 2:1 of basal to topdressed N. This result may be attributed to the lower N demand in the early growth stage of maize and the increased risk of leaching from a large amount of N fertilizer input. Kettering et al. (2013) indicated that applying fertilizer N with 3-4 splits according to a plant's N needs could reduce N leaching and increase the NRE.

Table 3. Grain yield, total N uptake and N recovery efficiency

Treatments	Grain Yield (t ha ⁻¹)		Total N uptake (kg ha ⁻¹)		NRE (%)	
	2014	2015	2014	2015	2014	2015
N0	8.6 c	3.6 c	125.2 d	50.1 d		
N1	12.7 b	12.1 b	218.7 c	169.1 c	41.6 c	52.9 c
N2	13.3 a	13.0 a	242.8 b	197.0 b	52.3 b	65.3 b
N3	13.5 a	13.2 a	255.0 a	221.9 a	57.7 a	76.4 a

Different lowercase letters within a column indicate significant differences ($p < 0.05$)

Conclusions

NH₃ flux was positively correlated with soil NH₄⁺-N content and was greatly affected by precipitation events. Applying N with two or three splits significantly increased the grain yield, total N uptake, and NRE of maize and significantly reduced NH₃ volatilization losses. The coupling effect of N fertilizer application and precipitation on NH₃ volatilization should be further investigated in the future.

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