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Fate of ^{15}N-labelled urea as affected by long-term manure substitution

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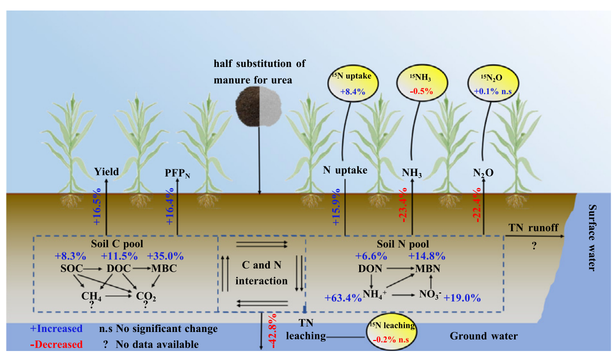
HIGHLIGHTS

- Long-term substitution (50 %) with N manure significantly increased NUE and reduced N losses.
- Manure substitution greatly suppressed ^{15}N leaching and $^{15}NH_3$ volatilization, while increasing $^{15}N_2O$ emissions.
- Manure substitution is beneficial for residue ^{15}N-urea kept in the soil nor uptake by plants.
- N surplus of CF is about twice as manure substitution (62.2 vs. 30.2 kg N ha^{-1}).

GRAPHICAL ABSTRACT



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ABSTRACT

Quantifying the fate of fertilizer nitrogen (N) is essential to develop more sustainable agricultural fertilization practices. However, the fate of chemical fertilizer N, particularly in long-term manure substitution treatment regimes, is not fully understood. The present study aimed to investigate the fate of ^{15}N-labelled urea in a chemical fertilizer treatment (CF, 240 kg ^{15}N ha^{-1}) and N manure 50 % substitution treatment (1/2N + M, 120 kg ^{15}N ha^{-1} + 120 kg manure N ha^{-1}) in two continuous crop seasons, based on a 10-year long-term experiment in the North China Plain (NCP). The results showed that manure substitution greatly enhanced ^{15}N use efficiency (^{15}NUE) (39.9 % vs. 31.3 %) and suppressed ^{15}N loss (6.9 % vs. 7.5 %) compared with the CF treatment in the first crop. However, the N_2O emissions factor in the 1/2N + M treatment was increased by 0.1 % (0.5 kg ^{15}N ha^{-1} for CF vs. 0.4 kg ^{15}N ha^{-1} for 1/2N + M) compared with the CF treatment, although N leaching and NH_3 volatilization rates decreased by 0.2 % (10.8 kg ^{15}N ha^{-1} for CF vs. 5.1 kg ^{15}N ha^{-1} for 1/2N + M) and 0.5 % (6.6 kg ^{15}N ha^{-1} for CF vs. 2.8 kg ^{15}N ha^{-1} for 1/2N + M), respectively. In which, only NH_3 volatilization presented significantly difference between treatments. It is important to note that in the second crop, the residual ^{15}N in soil (0–20 cm) remained mostly in the soil for the CF (79.1 %) and the 1/2N + M treatment (85.3 %), and contributed less to crop N uptake (3.3 % vs. 0.8 %) and leached losses (2.2 % vs. 0.6 %). This proved that manure substitution could enhance the stabilization of chemical N. These results suggested that long-term manure substitution effectively increases NUE, suppresses N loss, and improves N stabilization in soil, but negative impacts such as N_2O emissions due to climate change should be investigated further.

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1. Introduction

Nitrogen (N) underpins global food systems by boosting crop yields, ensuring soil fertility, and ultimately food security. Globally, chemical N fertilizer inputs in agricultural systems increased from 11 Tg (million ton) N year^{-1} in 1961 to 139 Tg N year^{-1} in 2020 (FAO, 2023), meeting global food demand but also leading to serious environmental pollution (e.g., eutrophication and air pollution) at regional and global scale (Ju et al., 2009; Yin et al., 2022). Measuring and monitoring the fate of fertilizer N and its impact on the environment (water and air phases) is required to guide improved N management strategies (Zhang et al., 2020).

It is well documented that the fate of fertilizer N is highly dependent on N application rates (Zhang et al., 2018b), application methods (Wu et al., 2017), and application time (Shi et al., 2012). The right application rate, application place, application time, and application method have been proven effective for higher NUE and lower N losses. Moreover, substituting chemical fertilizer with organic forms e.g. manure is also a common practice in agriculture and has promising effects on increasing NUE and mitigating fertilizer N losses. However, previous studies mostly focused on the effects of current management regimes but did not consider pre-existing management practices, which might induce great differences in soil fertility. Long-term application of inorganic or organic fertilizer leads to different soil fertility. In contrast to chemical fertilizers, long-term manure application generally improves soil organic carbon (SOC) contents (Gai et al., 2018), and enhances the activities of soil enzymes, i.e. urease, protease, cellulose, and β-glucosidase (Chang et al., 2007). The increased SOC results in increased soil total N (TN) and microbial biomass (Gai et al., 2019). The above factors play an important role in regulating microbial abundance and activity (Yang et al., 2023) and long-term manure application could greatly increase the mineralization-immobilization turnover (MIT) of N in the soil (Murphy et al., 2011; Li et al., 2020). A global meta-analysis showed that substituting manure for chemical N fertilizer significantly increased crop N uptake and significantly decreased reactive N (Nr) losses via NH_3 emission, N leaching, and N runoff, while it had no significant effect on the N_2O emission (Xia et al., 2017). However, a previous study in the North China plain (NCP) upland field found that the application of manure would enhance N_2O emission, and the increase of N_2O emission was much higher for long-term than short-term application of manure (Zhang et al., 2018a). Therefore, fertilizer NUE and losses can display highly varied patterns when applied to soils with variable background levels of soil fertility caused by various historical fertilization regimes (Liang et al., 2013).

The scientific background for increasing NUE and reducing Nr losses can be found by researching how fertilizer N behaves in soils with varying fertility (Zhang et al., 2021a). However, until now, previous ^{15}N tracer studies in agricultural systems have mainly focused on the effects of different fertilization treatments or soil fertility backgrounds on the fate of fertilizer N. Few studies have explicitly focused on the fate of fertilizer N in soils with long-term fertilization regimes. The NCP is one of the major areas for cereal production in China. Soils in the NCP are characterized by low fertility due to poor soil structure and low organic matter (OM) content (Gai et al., 2018). Under less fertile conditions, conventional farming practices only achieve a fertilizer N recovery of 27 % in the crop, 30 % of fertilizer N residue in the soil, and 47 % are lost to the environment (Ju and Zhang, 2017). Manure application has the capability to enhance fertilizer N recovery and reduce Nr losses (Xia et al., 2017; Gai et al., 2018). However, the fate of chemical fertilizer N in soils with long-term manure substitution practice is not clear, especially in the following crop season.

Therefore, a novel ^{15}N labelled experiment was conducted, including two treatments: chemical fertilizer treatment (CF, 100 % N from urea) and 50 % manure substitution treatment (1/2N + M, 50 % urea N plus 50 % manure N), based on a long-term field fertilization experiment established in 2007 in the NCP, while the N loss indicators were directly monitored as well. It was hypothesized that manure substitution would increase NUE and decrease N losses. Changes in soil properties caused by long-term manure substitution are responsible for higher NUE. However, the proportions of N loss reduction in N leaching, NH_3 volatilization and N_2O emission are inconsistent. The objectives were: (1) to investigate the effects of the long-term manure substitution for mineral fertilizers on maize production, NUE, and Nr losses; and (2) to identify the fate of fertilizer N in two continuous crop seasons under the two different long-term fertilization regimes. This would help further development of management decisions to increase NUE and reduce Nr losses in the NCP.

2. Materials and methods

2.1. Site description

The field experiment was conducted at the "Beijing Changping Soil Quality National Field Science Observation Research Station" (40.22°N, 116.23°E) in the NCP. This site is 43.5 m above sea level, has an average annual temperature of 11.5 °C, and mean rainfall of 625 mm. About 80 % of the precipitation occurs mostly from June to October each year, and irrigation occurs outside of this period. According to the FAO Taxonomy, the soil of the study area is Haplic Luvisol. This soil type is widely distributed in the NCP. At the start of the experiment, the bulk density (BD) of the top 20 cm soil was 1.32 g cm^{-3}, pH was 8.3, SOC was 7.9 g kg^{-1}, and TN was 0.76 g kg^{-1}. The crop used was spring maize, sown in mid to late May and harvested in early to mid-September each year.

2.2. Experimental design

The study was based on a long-term experiment with a randomized complete block experiment carried out in 2007 with three replicates. Each experimental plot (lysimeter) was an area of 1 m (width) × 2 m (length) × 1.2 m (depth). The bottom and sides of the plot were sealed by concrete. A 2-m underground monitoring station was built below the plot to facilitate weighing and monitoring the leachate. Leaching occurs mainly during the rainy season or irrigation; hence continuous seepage water collection was assured. The ^{15}N labelled experiment, including two treatments: CF (240 kg ^{15}N ha^{-1}) and 1/2N + M (120 kg ^{15}N ha^{-1} + 120 kg manure N ha^{-1}), were conducted during two continuous maize growing seasons of 2017–2018. In both treatments, urea was applied as base fertilizer (35.5 %) and topdressing (62.5 %) on 15th July 2017; and on 9th July 2018. The ^{15}N-labelled urea with an abundance of 20.15 % (Shanghai Research Institute of Chemical Industry, Shanghai, China) was only used in 2017, and unlabelled urea was used in the following crop season. The applied fertilizers were superphosphate (18 % P_2O_5) and potassium chloride (60 % K_2O), respectively. The swine manure used was air-dried, crushed, and analyzed as: OM content is 51.7 g kg^{-1}, TN content is 24 g kg^{-1}, P_2O_5 content is 16 g kg^{-1}, and K_2O content is 18 g kg^{-1}. The superphosphate, potassium chloride, and swine manure were applied before sowing. All fertilizers were distributed and immediately incorporated into the soil (0–20 cm depth). Detailed fertilization information for each treatment is provided in Table 1. Other management practices were in accordance with the local farming habits. Maize seed (cv. *Jingdan 28*) was

Table 1
Detailed fertilization information.

Treatments	Base urea (kg N ha^{-1})	Top-dressing urea (kg N ha^{-1})	Manure (kg N ha^{-1})	Total N (kg N ha^{-1})	P fertilizer (kg P_2O_5 ha^{-1})	K fertilizer (kg K_2O ha^{-1})
CF	90	150	0	240	120	180
1/2N + M	45	75	120	240	120	180

Abbreviations used in the table: N: nitrogen; P: phosphorus; K: potassium; CF: chemical fertilizer; 100 % chemical N application; 1/2N + M: 50 % chemical N + 50 % manure N application.

sown at a planting density of 60,000 ha^{-1} on 25th May, 2017 and 29th May 2018. The crop was harvested on maturity (2nd September 2017 and 14th September 2018). According to the automatic meteorological station located at the experimental field, the mean temperature was 30.9 °C. The total rainfall and irrigation water during the observation years were 526.0 mm and 258.7 mm, respectively.

2.3. Plant and soil sampling and measurement

The above ground crop samples from each treatment were harvested at maturity each year and separated into grain and straw. The straw was threshed, and the grain was weighed to achieve the biomass. The water content was measured by weighing the subsample of grain and straw, which were dried at 105 °C for 30 min, and then at 75 °C until they reached a constant weight. Grain yield is evaluated at 16 % water content in China. Thus, maize grain yields were calculated by multiplying dry grain yields by 116 %. In addition, N uptake by the crop was estimated from the N concentration in the dry matter and the mass of dry matter. The N content and ^{15}N abundance were measured using a stable isotope ratio mass spectrometer (Isoprime100, UK).

Soil samples (0–20 cm) were collected from each plot by a soil auger, and a composite soil sample was prepared by mixing the soil samples thoroughly. Soil NH_4^+-N and NO_3^--N were determined in 1 mol/L KCl extracts with a continuous flow analyzer (SEAL-AA3, Germany). NH_4^+-N and NO_3^--N in the extracts were further separated to determine ^{15}N abundance using the micro-diffusion method (Brooks et al., 1989). The rest of the soil was dried at room temperature and used to measure total N content and ^{15}N abundance using a stable isotope ratio mass spectrometer (Isoprime100, UK). Soil pH was measured with deionized water (1:2.5 soil/water). Soil TN and SOC were determined using the Kjeldahl digestion method (KDY-9830, Beijing), and the potassium dichromate oxidation method, respectively. The microbial biomass C (MBC) and microbial biomass N (MBN) were analyzed with the chloroform fumigation-extraction method (Gai et al., 2018), followed by analysis with an Automated TOC/TN Analyzer (3100/HT1300, Germany). The activities of β-D Glucosidase (BG) and *N*-acetylglucosidase (NAG) were measured using the method of Zhu et al. (2014).

2.4. Measurement of leaching and gas emission

The leachate was continuously collected, sampled and analyzed during rainfall and irrigation events. The concentration of TN and mineral N (NH_4^+-N and NO_3^--N) was analyzed using a potassium persulfate-ultraviolet spectrophotometer (UV-1780, SHIMADZU, China) and a continuous flow analyzer (SEAL-AA3, Germany), respectively (Li et al., 2018). The load of N leached was calculated by multiplying the volume of water by the N concentration.

Fluxes of NH_3 and N_2O were measured from each plot using active and passive closed chamber systems, respectively (Zhang et al., 2018a). For measuring N_2O fluxes, the chamber system consisted of a plastic bottom frame (50 cm × 25 cm × 35 cm) and a top chamber (50 cm × 25 cm × 120 cm). Gas samples were collected using a 100 mL syringe at 0, 20, and 40 min intervals and the samples were stored in vacuum bags. The collected gas samples were analyzed using a Shimadzu GC-2010 plus gas chromatograph (GC) equipped with an electron capture detector (ECD) for N_2O measurement. Gas samples were taken every second day for the first 15 days after fertilizer application and then once per week thereafter. All passive chambers were periodically calibrated by collecting known volumes of N_2O standards injected under the enclosures, and 98–101 % recoveries were obtained before field use.

For measuring NH_3 fluxes, an active gas chamber system powered by a battery was used to collect gas samples. The chamber was cylindrical and consisted of a plastic frame (250 mm inner diameter, 350 mm height) and a top chamber (260 mm inner diameter, 150 mm height). The chamber was placed between maize rows before basal fertilization. NH_3 volatilization from the soil surface was flushed by the NH_3-free air and bubbled into the impinger containing 40 mL of 0.05 mol/L sulfuric acid solution to trap emitted NH_3. The trapped NH_3 was measured using an XY-2 sampler connected to the continuous-flow analyzer (AA3-Germany), and the NH_3 fluxes were calculated based on the flow rate and collection time per unit area of soil. All active chamber systems were periodically calibrated before field use by measuring the recovery of a spike of NH_3 produced by reacting a known concentration of NH_4OH with NaOH under complete enclosure for 2 h. Air and soil (0–10 cm) temperatures were recorded using temperature probes, and soil moisture levels were determined using a TDR 100 soil moisture meter (Spectrum Technologies, Inc., Item # 6440FS).

The atom% ^{15}N of the trapped N_2O and NH_3 was measured using a stable isotope ratio mass spectrometer (Isoprime100, UK). All operation procedures were conducted sequentially from lower to higher atom% ^{15}N to avoid cross-contamination.

2.5. Calculations and statistical analysis

The crop NUE, partial factor productivity from applied N (PFP_N, kg kg^{-1}), and soil surface N balance were calculated as follows:

$$\text{NUE }(\%) = \text{N uptake/N application rate} \times 100$$

$$\text{PFP}_\text{N}\ \left(\text{kg kg}^{-1}\right) = \text{Grain yield/N application rate}$$

$$\text{Soil surface N surplus}\ \left(\text{kg N ha}^{-1}\right) = \text{total N input} - \text{total N uptake};$$

where total N input includes N from fertilizer, atmospheric deposition, and biological fixation.

The percentage of total plant ^{15}N, and soil residual ^{15}N derived from ^{15}N fertilizer was calculated as follows:

$$\%\text{Ndff}_{\text{crop (soil)}} = (\text{c} - \text{b})/(\text{a} - \text{b}) \times 100$$

where c, a, b is the ^{15}N abundance of the crop (soil), fertilizer (20.25 %), and natural ^{15}N abundance, respectively.

Crop ^{15}N uptake, residual ^{15}N in the soil, and ^{15}N losses were calculated as follows:

$$^{15}\text{N uptake}\ \left(\text{kg N ha}^{-1}\right) = \%\text{Ndff}_{\text{crop}} \times \text{crop N uptake}$$

$$^{15}\text{NUE }(\%) = {}^{15}\text{N uptake}/\,{}^{15}\text{N application rate}$$

$$\text{Residual}^{15}\text{N in soil}\ \left(\text{kg N ha}^{-1}\right) = \%\text{Ndff}_{\text{soil}} \times \text{bulk density} \times \text{thickness} \times \text{soil}^{15}\text{N content} \times 100$$

$$^{15}\text{N losses}\ \left(\text{kg N ha}^{-1}\right) = {}^{15}\text{N leaching} + {}^{15}\text{NH}_3\text{ volatilization} + {}^{15}\text{N}_2\text{O emission}$$

$$\text{Irrigation N}\ \left(\text{kg N ha}^{-1}\right) = \text{Irrigation amount} \times \text{TN content}$$

The atmospheric N deposition flux used in the budget was 32.5 kg N ha^{-1} per year, an averaged value during maize season in the NCP (Gai et al., 2018), irrigation N was calculated by multiplying the irrigation amount with the TN content of the irrigation water, and the non-symbiotic N fixing for maize was 5 kg N ha^{-1} (Bouwman et al., 2013; Herridge et al., 2008).

Microsoft Excel 2016 was used for data processing and graphs. Data were presented as means ± standard errors of the three replicates. Data were statistically analyzed using SPSS software (version 19.0). The significant differences between the treatments were determined using one-way ANOVA with Tukey's test for multiple comparisons at a *P*-value of 0.05.

3. Results

3.1. Changes in soil properties under the long-term field experiment

Compared to long-term application of chemical fertilizer N (CF treatment), long-term 50 % manure substitution (1/2N + M treatment) significantly increased the content of TN, SOC, NO_3^--N, NH_4^+-N, MBC and MBN by 33.3 %, 8.3 %, 19.0 %, 63.4 %, 35.0 % and 14.5 %, respectively. It also slightly reduced soil pH (8.2 vs. 8.3) and had no effect on BD (1.3 vs. 1.3). Furthermore, long-term 50 % manure substitution also reduced the abundance of the ammonia-Oxidizing Archaea (AOA) and ammonia-Oxidizing Bacteria (AOB) gene by 5.1 % and 32.1 % and also increased the enzyme activity of BG and NAG by 16.3 % and 7.0 %, respectively (Table 2).

3.2. Crop N uptake and chemical N losses

The significant variation in grain and straw biomass during the experimental period is presented in Fig. 1a. Manure substitution enhanced the crop yield significantly. During the two crop seasons (2017 and 2018), the maize yields in the 1/2N + M treatment were significantly increased by 19.5 % and 13.4 % compared to the CF treatment (8.76 vs. 7.33 and 8.47 vs. 7.47 t ha^{-1}), respectively; the straw biomass was found to have increased by 31.3 % and 8.5 % in the 1/2N + M treatment compared with the CF treatment (7.71 vs. 5.87 and 11.49 vs. 10.59 t ha^{-1}), respectively. Thus, the crop N uptake and PFP_N were improved by manure substitution (Fig. 1b and c). The crop N uptake of grain and straw in the 1/2N + M treatment (258.0 kg N ha^{-1}) was notably higher than in the CF treatment (197.7 kg N ha^{-1}) in 2017. Despite the fact that there was no significant difference between the two treatments in 2018, the crop N uptake in the 1/2N + M treatment still increased by 3.6 % compared to the CF treatment. For the 1/2N + M treatment, PFP_N reached 36.5 and 35.3 kg^{-1} in 2017 and 2018, both markedly increased by 19.5 % and 13.4 %, respectively, compared to the CF treatment.

TN losses in each loss path involving TN leaching, NH_3 volatilization, and N_2O emission showed remarkable differences between treatments (Fig. 1d, e, f). In the CF treatment, TN losses, covering 76.3 kg N ha^{-1} through TN leaching, 8.0 kg N ha^{-1} through NH_3 volatilization, and 1.4 kg N ha^{-1} through N_2O emission, were all significantly higher than in the 1/2N + M treatment, in which 42.0 kg N ha^{-1} were lost by TN leaching, 6.0 kg N ha^{-1} by NH_3 volatilization and 1.2 kg N ha^{-1} by N_2O emission. In summary, manure substitution reduced 45.0 % of TN losses compared with the chemical fertilizer treatment.

3.3. The fate of ^{15}N-labelled urea in the spring maize system

It is widely known that the fate of fertilizer N in the fertilizer-soil-crop-environment continuum includes crop uptake, soil storage, and environmental losses in various ways. In this study, the fate of ^{15}N was compared in different treatments by analyzing the proportion of ^{15}N that was absorbed by the crop, resided in the soil, and was lost to the environment of the applied ^{15}N. Fig. 2 shows that manure substitution boosted crop ^{15}N uptake while it decreased the residual ^{15}N in the soil and the chemical fertilizer ^{15}N loss, although there was no significant difference in the total fertilizer ^{15}N losses. In the first crop season, the crop ^{15}N uptake, the loss of ^{15}N to the environment, and the residual ^{15}N in the 0–20 cm soil were 39.9 %, 39.8 %, and 20.3 % in the 1/2N + M treatment; while they were 31.5 %, 41.2 % and 27.3 % in the CF treatment. Based on the N flux balance, about 33 % of fertilizer ^{15}N was still not detected in either treatment, which mainly stored in the 20–90 cm soil profile and also partially lost by N_2.

In terms of fertilizer ^{15}N losses, manure substitution decreased the TN leaching and NH_3 volatilizations, but increased N_2O emissions. For the CF treatment, the fertilizer ^{15}N losses were 17.9 kg N ha^{-1}, of which the TN leaching, NH_3 volatilizations, and N_2O emissions were 10.8, 6.6, and 0.5 kg N ha^{-1}, accounting for 4.5 %, 2.8 %, and 0.2 % of the applied ^{15}N fertilizer, respectively. For the 1/2N + M treatment, fertilizer ^{15}N losses were 8.3 kg N ha^{-1}, of which 5.1 kg N ha^{-1} were lost through TN leaching, 2.8 kg N ha^{-1} through NH_3 volatilizations and 0.4 kg N ha^{-1} through N_2O emissions, accounting for 4.3 %, 2.3 % and 0.3 % of the applied ^{15}N fertilizer, respectively. This implied that manure substitution could suppress the N leaching and NH_3 volatilizations, but would increase N_2O emissions, so

Table 2
Soil physical, chemical and biological properties in 2017 after 10 years of fertilization.

Treatment	pH	Total N ($g\ kg^{-1}$)	Organic carbon ($g\ kg^{-1}$)	Bulk density ($g\ cm^{-3}$)	NO_3^--N ($mg\ kg^{-1}$)	NH_4^+-N ($mg\ kg^{-1}$)	MBC[§] ($mg\ kg^{-1}$)	MBN ($mg\ kg^{-1}$)	AOA (10^6 copies g^{-1} dry soil)	AOB (10^6 copies g^{-1} dry soil)	BG ($nmol\ g^{-1}\ h^{-1}$)	NAG ($nmol\ g^{-1}\ h^{-1}$)
CF[‡]	8.3 ± 0.0a[†]	0.9 ± 0.1a	9.6 ± 0.3a	1.3 ± 0.0a	13.7 ± 1.1a	4.1 ± 0.4a	186.0 ± 9.9a	26.4 ± 0.9a	45.1 ± 3.9a	13.1 ± 3.0a	197.9 ± 13.4a	423.2 ± 7.0a
1/2N + M	8.2 ± 0.0a	1.2 ± 0.0b	10.4 ± 0.2b	1.3 ± 0.0a	16.3 ± 1.7b	6.7 ± 0.1b	251.0 ± 27.0b	30.3 ± 0.8b	42.8 ± 8.3a	8.9 ± 3.6a	230.2 ± 5.8b	453.0 ± 19.8a

† Number represents mean ± standard error (n = 3), the different lowercase letters indicate significant differences ($P < 0.05$) between treatments.
‡ N: nitrogen; CF: chemical fertilizer; 100 % chemical N application; 1/2N + M: 50 % chemical N + 50 % manure N application.
§ MBC: microbial biomass carbon; MBN: microbial biomass nitrogen; AOA: ammonia-Oxidizing Archaea; AOB: ammonia-Oxidizing Bacteria; BG: β-D-glucosidase; NAG: *N*-acetylglucosaminidase.

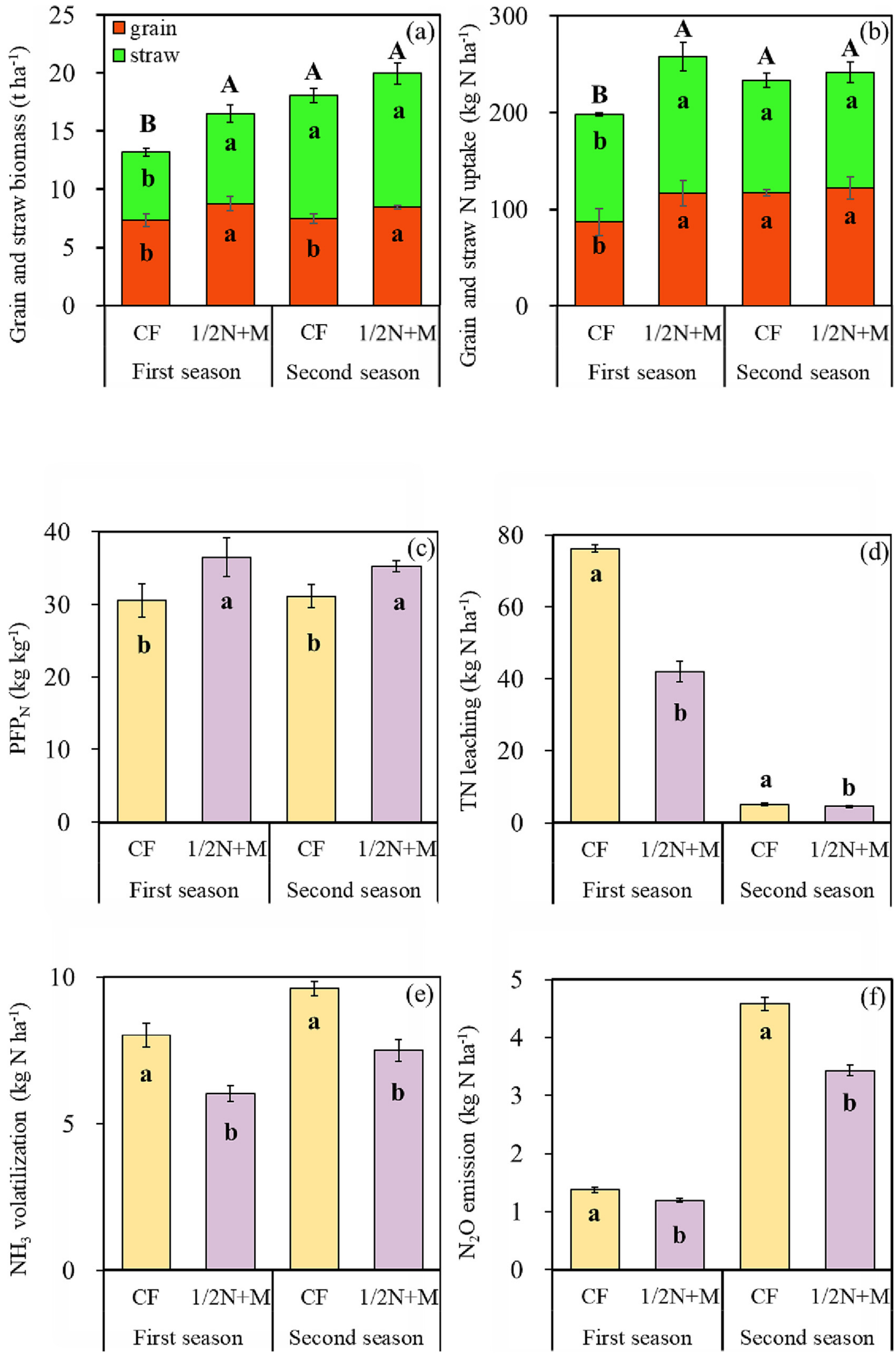

Fig. 1. Maize yield, N uptake and fertilizer N losses. CF and 1/2N + M refer to chemical fertilizer treatment and half manure substitution treatment, respectively. PFP_N is the partial factor productivity from applied N. Error bars represent standard errors (n = 3), lowercase letters compare the parameters of the corresponding legend among treatments, uppercase letters compare the parameters of the corresponding whole column among treatments, the different lowercase or uppercase letters indicate significant differences ($P < 0.05$) between treatments.

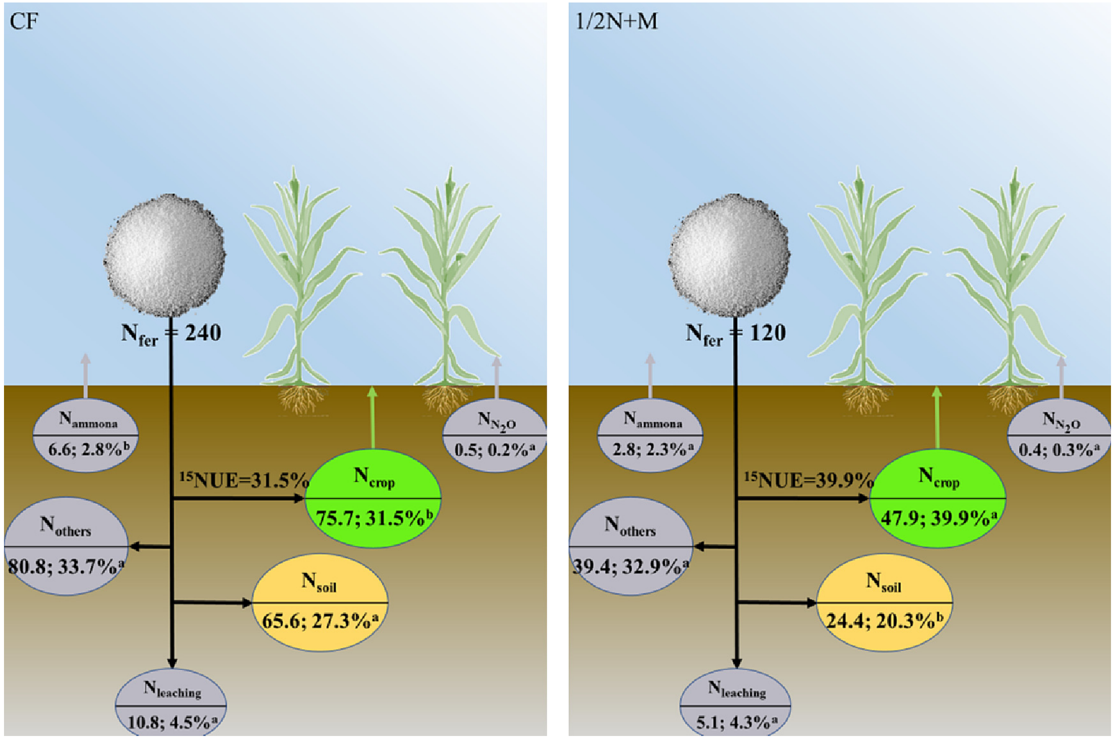

Fig. 2. The fate of ^{15}N in the fertilizer-soil-crop-environment continuum under the CF treatment and 1/2N + M treatment. Data were derived from the first maize season (2017), and the unit for the numbers is kg N ha^{-1}. N_{fer}, N_{crop} and N_{soil} represent ^{15}N rate, ^{15}N uptake by crop, residual ^{15}N in 0–20 cm soil. $N_{ammonia}$, N_{N2O}, $N_{leaching}$, and N_{others} represent ^{15}N losses through NH_3 volatilization, N_2O emissions, TN leaching and undetected ^{15}N. The percentage indicates the proportion of each ^{15}N fate in the applied fertilizer N; different lowercase letters indicate significant differences ($P < 0.05$) between treatments.

the trades among N losses should be considered when adopting a regime of manure application.

In the following season, the residual ^{15}N in soil (0–20 cm) was mainly stored in soil (Table 3). It was notable that manure substitution treatment stored more residual ^{15}N in the 0–20 cm soil (85.3 %) compared with the CF treatment (79.1 %). The contributions of residual ^{15}N in soil (0–20 cm) to crop N uptake (0.8 % vs. 3.3 %) and N leaching loss (0.6 % vs. 2.2 %) in the 1/2N + M treatment were significantly less than those in the CF treatment. There were no NH_3 volatilizations or N_2O emissions detected from the residual soil ^{15}N in the second crop season. Downward movement into deep soil dominated the loss of residual ^{15}N, accounting for 15.3 % and 13.5 % of residual ^{15}N in the CF and 1/2N + M treatments, respectively. These results further proved that long-term manure substitution would effectively improve chemical fertilizer N stabilization and decrease its mobility in soil.

3.4. Surface N balance in spring maize system

In farmland biogeochemical cycles, N input flows mainly include atmospheric N deposition, fertilizer N application, and biological N fixation, while N output flows include leaching, NH_3 volatilization, crop uptake, and N loss in nitrification and denitrification. As shown in Table 4, the surface N surplus in the 1/2N + M treatment was 24.2 kg N ha^{-1}, while in the CF treatment, it was 79.9 kg N ha^{-1} in 2017. Similar to the trend of 2017, the soil surface N balance was 36.1 kg N ha^{-1} and 44.5 kg N ha^{-1} in 2018 for the 1/2N + M treatment and CF treatment, respectively. Interestingly, the reductions in the surface N surplus induced by manure substitution were greatly different between 2017 (55.7 kg N ha^{-1}) and 2018 (8.4 kg N ha^{-1}). This result suggested that the contribution of manure substitution to the N transportation was affected by the regional climate, which would affect crop yield, crop N uptake ability, etc. Thus, it can be seen that manure substitution was beneficial for the N surface balance, but its contribution ability is not constant because of the annual variations.

3.5. Pearson correlations and structural equation model (SEM)

Crop N uptake showed positive correlations with manure substitution and background SOC levels and MBC, MBN, NO_3^--N (Fig. 3). The volatilized N_2O revealed a stronger correlation with uptake, SOC, MBN, and NO_3^--N. The SEM model showed that manure application significantly increased SOC levels with a factor loading of 0.59**. Higher SOC levels resulted in higher MBC and MBN with greater factor loadings of 0.91** and 0.82**, which indirectly improved crop N uptake by increasing NH_4^+-N and NO_3^--N (Fig. 4). In addition, manure application significantly increased

Table 3
Fate of residual fertilizer ^{15}N in the second crop trial.[§]

Treatments	Crop uptake[¶]		Residue in soil (0–20 cm)		Leached losses		Others	
	Amount (kg N ha^{-1})	Proportion %	Amount (kg N ha^{-1})	Proportion %	Amount (kg N ha^{-1})	Proportion %	Amount (kg N ha^{-1})	Proportion %
CF[‡]	2.2 ± 0.3	3.3 ± 0.5a[†]	51.9 ± 4.5	79.1 ± 6.7a	1.5 ± 0.1	2.2 ± 0.1a	10.0 ± 4.1	15.3 ± 6.3a
1/2N + M	0.2 ± 0.0	0.8 ± 0.2b	20.7 ± 4.1	85.3 ± 3.7a	0.1 ± 0.0	0.6 ± 0.1b	3.3 ± 0.7	13.5 ± 3.6a

† Number represents mean ± standard error (n = 3), the different lowercase letters indicate significant differences ($P < 0.05$) between treatments.
‡ N: nitrogen; CF: chemical fertilizer; 100 % chemical N application; 1/2N + M: 50 % chemical N + 50 % manure N application.
§ The urea with ^{15}N labelled was only used in 2017 while the N fertilizer applied in 2018 was the urea without ^{15}N labelled.
¶ Crop uptake is the sum of the N assimilated by maize grain and straw.

Table 4
Soil surface N surpluses in spring maize trial (kg N ha^{-1}).

Year	Treatments	Fertilizer N	Deposition N	Irrigation N	Biological N fixation	Crop N uptake[b]	N surplus[c]
2017	CF[a]	240	32.5	0.1	5.0	197.7	79.9
	1/2N + M	240	32.5	0.1	5.0	253.4	24.2
2018	CF	240	32.5	0.1	5.0	233.1	44.5
	1/2N + M	240	32.5	0.1	5.0	241.5	36.1

[a] N: nitrogen; CF: chemical fertilizer; 100 % chemical N application; 1/2N + M: 50 % chemical N + 50 % manure N application.
[b] Crop aboveground N uptake is the sum of the N assimilated by maize grain and straw.
[c] Soil surface N surplus was calculated by subtracting the total N uptake from N inputs.

NH_4^+-N in soil with high factor loadings of 0.27***, and NH_4^+-N can directly reduce crop N uptake with greater factor loadings of −1.02**, and it also indirectly improved crop N uptake by converting it into NO_3^--N. The data fit well with the hypothetical model as indicated by the selection criteria such as df, P, cfi, rmsea, and srmr.

4. Discussion

4.1. Influence of manure substitution on ^{15}N use efficiency

The present research indicated that equal N substitution of manure significantly increased grain yield and chemical N utilization efficiency (Fig. 1), which is in accordance with previous studies (Li et al., 2022; Xia et al., 2017). The organic fertilizer has been shown to promote the decomposition of organic substrates and SOC by increasing microbial biomass and microbial activity to produce more mineral N for the demand of plants, thus enhancing crop uptake of N (Ma et al., 2018; Mcgill and Cole, 1981; Moreau et al., 2019). On the other hand, applying organic fertilizer can boost root growth and density, increase root biomass, and prolong rooting depth (Ashraf et al., 2004; Kurdali, 2004), hence increasing plant absorption of soil N (Azam and Mulvaney, 1993). The greater synchronization of N supply and crop demand in the manure substitution treatment might contribute to the increase of grain yield and crop N uptake in the manure substitution treatment. Moreover, long-term application of manure resulted in a slight decrease in soil pH from 8.3 to 8.2 (Table 2). Weak acidification has been proved to lead to a more open N cycle (Wang et al., 2023). This outcome is probably connected to the higher mineralization-immobilization turnover (Dai et al., 2017; Wang et al., 2015). The higher ^{15}N use efficiency in the manure substitution treatment also proved the contribution of manure to the improvement of grain yield and crop N uptake.

Surprisingly, substitution of manure with 50 % N dramatically increased the crop NUE compared with the chemical fertilization (39.9 % vs. 31.5 %) in the first crop season; while for the following crop season, the ^{15}N use efficiency in the 1/2N + M treatment (0.8 %) was lower than that of the CF treatment (3.3 %) (Fig. 2). In this investigation, the long-term manure application had significantly higher soil SOC, TN, NO_3^--N, NH_4^+-N concentrations than that in the CF treatment (Table 2). After the application of organic fertilizers, the availability of C increased, causing a large microbial demand for N, thus increasing the immobilization rates of NH_4^+-N (Chapman, 1997; Khalil et al., 2005). The increasing NH_4^+-N immobilization rate caused by organic fertilizer application indicated that additional N could be immobilized into the soil organic N pool, which could subsequently be available through remineralization (Manzoni et al., 2017; Mooshammer et al., 2014). Over time, the slow release of sequestered N can sustainably support the need of both microbes and plants, leading to an eventual increase in N mineralization (Wang et al., 2015; Wyngaard et al., 2016).

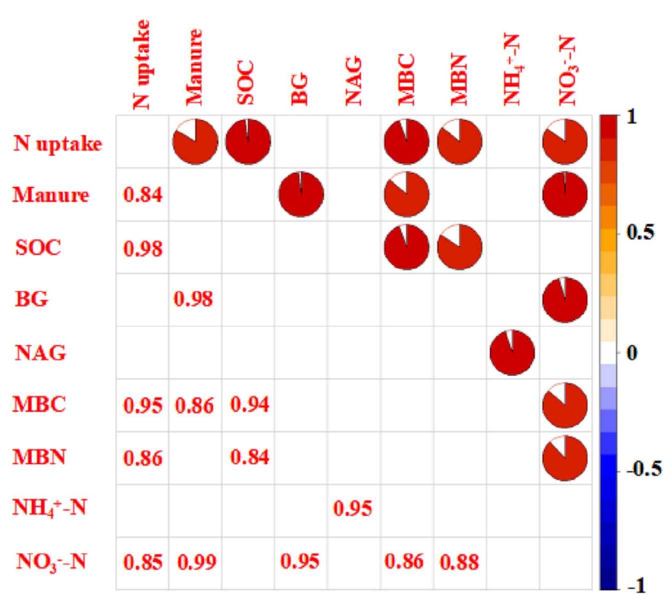

Fig. 3. The Pearson's correlation coefficients for N uptake, application of manure, background soil organic carbon, β-D glucosidase (BG) and *N*-acetylglucosidase (NAG) enzyme activity, soil microbial biomass carbon, soil microbial biomass N, NH_4^+-N and NO_3^--N contents. The color gradient is proportional to the Pearson's correlation coefficient. Red and blue colors denote positive and negative relationships, respectively. The shown figures indicate significant differences ($P < 0.05$).

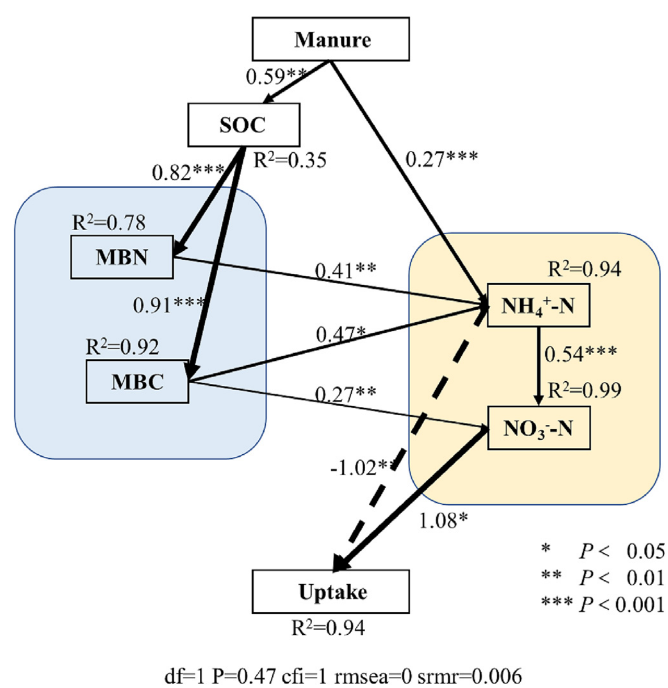

Fig. 4. Structural equation model for N uptake and soil microbial biomass carbon, soil microbial biomass N, NH_4^+-N, NO_3^--N contents as affected by application of manure and background soil organic carbon. The number adjacent to arrow line is factor loading that shows the variance explained by the variable, and the width of the line is proportional to the strength of factor loading. + and − indicate the negative and positive effect, respectively. Significant paths are marked with * ($P < 0.05$), ** ($P < 0.01$) or *** ($P < 0.001$). Only significant explanatory and necessary variables with high factor loading were considered in SEM analysis.

This is also in line with the results of the model analysis. The SEM indicated that manure application could not only sustain microbial and plant requirements through the progressive breakdown of its organic substrates to produce NH_4^+-N directly, but also promote the microbial immobilization of bioavailable N by increasing the supply of organic carbon (Manzoni et al., 2017; Mooshammer et al., 2014). The immobilized N would be released throughout the crop growing season, thereby increasing N uptake and crop yield (Fig. 4). It should be noted that due to the long-term manure application treatment, SOC levels were not only influenced by the current crop season manure application but also by long-term treatments in the past, which explains the low explanation level ($R^2 = 0.35$) of manure application on SOC levels.

In the second year, the lower residual ^{15}N use efficiency in the 1/2N + M treatment was also associated with the increased N sequestration capacity of the soil by manure application, this can be explained in two ways. First, the residual ^{15}N leaching losses were significantly higher in the CF treatment than in the 1/2N + M treatment. This indicates that the residual ^{15}N in the CF treatment was not stable compared to the 1/2N + M treatment and had a significant trend of downward movement. This was backed up by a lower proportion of residual ^{15}N in the CF treatment that remained in the surface soil and a higher proportion that entered the deeper soil layer compared to the 1/2N + M treatment. Additionally, in the present study, the long-term manure application treatment had significantly higher soil SOC, TN, NO_3^--N, NH_4^+-N, MBC, and MBN levels than the CF treatment (Table 2). It is commonly believed that if SOC concentrations are higher, inorganic N is easily transformed to organic N, which enhances soil retention of N (Dai et al., 2017; Lu et al., 2018). The rise of MBC and MBN reflects an improved ability of N retention by microbial communities in the manure substitution treatment. This was supported by dramatically increased β-D Glucosidase and *N*-acetylglucosidase enzyme activity, and the abundance of the AOB gene (Table 2).

4.2. Chemical N fate in the current crop and subsequent impacts

In the first crop season, TN leaching was significantly reduced by 81.6 % in the 1/2N + M treatment compared to the CF treatment. In the second crop season, total N leaching was not significantly different between the treatments. These results were inconsistent with the meta-analysis results of Xia et al. (2017), who found that substituting different organic manures for compound fertilizer decreased N leaching by 28.9 %. In addition, it was noted that TN leaching was significantly higher in 2017 than in 2018 for both treatments, which was caused by the high interannual differences in precipitation (495.8 mm in 2017 vs. 221.0 mm in 2018) and the extreme precipitation events that occurred in 2017 (Fig. S1). Another possible reason is the increasing availability of carbon sources, which may encourage the dissimilatory NO_3^--N reduction to NH_4^+-N after manure application. Gao et al. (2010) showed that the N leaching was dominated by NO_3^--N during the annual cycle of wheat-corn rotation. Therefore NO_3^--N isomerization to NH_4^+-N enhances soil N retention and minimizes NO_3^--N runoff and leaching (Wang et al., 2015). Furthermore, ^{15}N leaching in the CF treatment accounted for only 14.2 % of TN leaching, indicating that heavy precipitation promotes soil N leaching. Compared to the CF treatment, NH_3 volatilization was significantly reduced by 33.0 % and 28.4 % in the first and second crop, respectively, in the 1/2N + M treatment. This result is consistent with Xia et al. (2017), who concluded through a meta-analysis that manure substitution was able to reduce NH_3 volatilization by 26.8 %. Among them, NH_3 volatilization from ^{15}N in the CF treatment accounted for 82.3 % of TN volatilization, but only for 46.4 % in the 1/2N + M treatment, indicating that NH_3 volatilization in the conventional fertilization treatment mainly came from the chemical fertilizer used in the current season, and manure also produced part of the NH_3 volatilization loss in the manure substitution treatment. N_2O emissions also showed a significant reduction in the manure substitution treatment. N_2O emissions were significantly reduced by 15.1 % and 33.5 % in the first and second crop seasons of the 1/2N + M treatment compared to the CF treatment, which was consistent with previous studies (Liu et al., 2015a; Xia et al., 2017; Ozlu and Kumar, 2018). It could be due to increased immobilization of mineral N by microbes after manure application (Ma et al., 2018), which reduces the substrate supply for nitrification and denitrification (Wang et al., 2015; Zhou et al., 2016) and promotes plant uptake of inorganic N. This is proven by the rise in MBC and MBN, as well as the substantial increase in BG and NAG enzyme activity and the abundance of AOA and AOB gene (Table 2).

Interestingly, a large interannual variation in N_2O emissions between 2017 and 2018 was noted, this might be caused by interannual differences in precipitation. Hou et al. (2018) showed that N_2O emissions were positively correlated with NO_3^-- N content and soil water content. Wang et al. (1995) also showed that the peak of N_2O emissions during the maize growing season occurred mainly after fertilization, irrigation, or rainfall. Song et al. (2019) demonstrated that oxygen is critical in regulating N_2O production in well-structured upland agricultural soil. Irrigation or precipitation reduced diffusion rates of O_2 in the water phases (Neira et al., 2015), directly causing O_2 depletion and leading to anoxic conditions that promoted nitrifier denitrification, coupled nitrification denitrification, or heterotrophic denitrification. Due to the lack of real-time monitoring data on soil water content and O_2 concentration, the reasons for the significant interannual differences in N_2O emissions remain unclear. Although the precipitation in the test area was significantly higher in 2017 than in 2018, precipitation in 2017 was concentrated in several extreme precipitation events, and the rest of the precipitation events generally had less precipitation. There were no extreme precipitation events in 2018, but its precipitation distribution was more uniform and denser. Therefore, it was hypothesized that due to the dense and uniform precipitation events in 2018, the soil was in a longer period of hypoxia, leading to a significant enhancement of N_2O. This highlights the link between climate and management factors on N_2O production.

There was no significant difference in ^{15}N losses in the first crop season between the CF and 1/2N + M treatments. This finding is consistent with the results reported by Zhang et al. (2021a). There was no significant difference in the losses between the two treatments in the first season. Among them, the labelled urea applied in the CF treatment had an NH_3 emission factor of 2.8 % and N_2O volatilization factor of 0.2 %. These two values decreased to 2.3 % and increased to 0.3 % in the 1/2N + M treatment, respectively. This indicates that the manure substitution treatment reduced NH_3 emissions while increasing N_2O volatilization. NH_3 volatilization is the result of NH_3 transport from the surface of the ammoniacal solution to the atmosphere (Ju and Zhang, 2017). The environmental conditions of simultaneous rain and heat during the growing season of maize in the NCP can promote manure decomposition and subsequently enhance adsorption of NH_4^+ and NH_3 through the addition of particles or colloids (Xu et al., 2022). In addition, the swine manure used was air-dried, crushed, which also reduced the NH_3 emission potential (Sommer and Hutchings, 2001). Wei et al. (2023) demonstrated that O_2 dynamics were the proximal determining factor to matrix N_2O concentration. The application of manure increased N_2O emissions from fertilizer N may be related to the transient N_2O accumulation and emissions caused by the anoxic conditions around the applied manure in soils (Markfoged et al., 2011). The NH_3 emission factors of synthetic fertilizers mostly ranged from 5 % to 15 % (Fu et al., 2020; Guo et al., 2020; Zhang et al., 2021b), and N_2O volatilization factors mostly ranged from 0.9 % to 1.4 % (Hoben et al., 2011; Liu et al., 2015b; Wang et al., 2018). The lower NH_3 volatilization factor may be related to the immediate incorporation of fertilizers into the soil (0–20 cm depth) by turning the soil immediately after spreading fertilizers during the basal fertilization period and wet season during the topdressing period (which can wash urea into the soil quickly). In addition, the lower average annual temperature in the NCP compared to South China may reduce its NH_3 emission potential (Fu et al., 2020). The lower N_2O emissions factor may be related to the amount of N application. N application is the main factor influencing N_2O emissions during the same period of rain and heat in the maize growing season (Liu et al., 2015b). Moreover, 240 kg N ha^{-1} is a reasonable rate of N application in the study area (Song et al., 2019), and appropriate N application positively reduces N_2O emissions. Manure substitution slightly reduced the percentage of ^{15}N leaching losses (4.5 % vs. 4.3 %). Leaching in dryland cropping systems is mainly caused by precipitation, so there may be a large interannual variation even in the same treatment. Also, manure substitution significantly increases soil water retention, water-stable aggregates (>0.25 mm) fraction and cation exchange capacity of the soil, which facilitates the adsorption of soil mineral N and thus reduces N leaching (Shisanya et al., 2009; Alidad et al., 2012; Xia et al., 2017).

4.3. Future N management

The N surplus at the soil surface is typically used to measure N losses. N surplus criteria might be considered the maximum N loss permitted in a particular cropping scheme (Ju and Gu, 2017; Zhang et al., 2019). In the long term, if the N surplus is more than a benchmark, it will be anticipated that N release to the environment will be intolerable. However, if the balance decreases below zero, there is a possibility for soil N mining (EU Nitrogen Expert Panel, 2016). On a regional level, it has been suggested that the NCP summer maize should have a N surplus standard of 80 kg N ha^{-1} (Ju and Gu, 2017; Zhang et al., 2019). The CF and 1/2N + M treatments in this investigation had N surpluses of 62.2 kg N ha^{-1} and 30.2 kg N ha^{-1}, respectively, less than the suggested N surplus standard. This does not imply that all treatments include mining soil N, though. The 80 kg N ha^{-1} of the N standard is an achievable goal for improving the N management of present conventional practices with a substantial N surplus, and advancements might further reduce it in fertilization methods and agronomic management (Zhang et al., 2019). All the treatments in the current study got the optimal N management; for spring maize in the study region, 240 kg N ha^{-1} of fertilizer N was an appropriate N rate (Song et al., 2019). The N losses were relatively minimal because fertilizer was provided at the proper time and was well integrated into the soil by irrigation or plowing (Huang et al., 2013, 2017; Qiu et al., 2012; Song et al., 2019). Overall, 50 % manure substitution was a viable fertilization strategy since it was significantly involved in improved crop yields while increasing soil C inputs, thus preventing soil fertility from being overexploited, whereas the chemical fertilizer only application ran the risk of suffering significant N losses.

5. Conclusions

This study was able to clearly show that 50 % manure substitution could significantly increase the above-ground biomass and crop N uptake, enhance soil ability to sequester N, and reduce the loss of fertilizer N. 50 % manure substitution was a reasonable fertilization measure, close to the amount of applied N required for high yield and soil N balance calculated by the soil N balance method, while conventional fertilization had the risk of high N losses. These results highlight the importance of manure application in soil. Surprisingly, the ^{15}N use efficiency in the 1/2N + M treatment was significantly higher than the CF treatment in 2017. While in 2018, the utilization of residual ^{15}N by crop in the 1/2N + M treatment was notably lower compared to the CF treatment. This may be attributed to the increased N sequestration capacity of the soil by manure application. Future studies should focus on the use efficiency of residual ^{15}N in subsequent years, fate, and form of existence in the soil. In addition, the reasons for the decreased residual ^{15}N use efficiency under manure application needs further study with specific focus on the effects caused by different manure types (fresh slurry/liquid versus composted manure) and different substitution ratios.

CRediT authorship contribution statement

Fuyue Dai: Data curation, Investigation, Methodology, Writing – original draft. **Bingqian Fan:** Conceptualization, Writing – review & editing, Validation. **Jungai Li:** Software, Formal analysis, Writing – review & editing. **Yitao Zhang:** Writing – review & editing, Visualization. **Hongyuan Wang:** Conceptualization, Funding acquisition, Project administration, Resources, Supervision. **Zhen Wang:** Writing – review & editing, Visualization. **Muhammad Amjad Bashir:** Writing – review & editing, Validation. **Golnaz Ezzati:** Writing – review & editing, Visualization. **Limei Zhai:** Writing – review & editing, Validation. **Hong J. Di:** Writing – review & editing, Visualization. **Hongbin Liu:** Conceptualization, Funding acquisition, Project administration, Writing – review & editing.

Data availability

Data will be made available on request.

Declaration of competing interest

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

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Appendix A. Supplementary data

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