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Model the Relationship of NH_3 Emission with Attributing Factors from Rice Fields in China: Ammonia Mitigation Potential Using a Urease Inhibitor

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Abstract: Substantial ammonia (NH_3) losses from rice production result in poor nitrogen (N) use efficiency and environmental damage. A data synthesis using the published literature (127 studies with 700 paired observations), combined with an incubation experiment using 50 paddy soils from across China, were conducted to improve the current understanding of the NH_3 loss from paddy rice and its drivers. The efficacy of the urease inhibitor Limus® for reducing NH_3 losses was also evaluated. The mean loss of N, through NH_3 volatilization, was 16.2% of the urea-N applied to paddy rice. The largest losses were from double rice cropping systems, and losses increased with the N application rate, surface application of N, unstable N types (ammonium bicarbonate and urea), and high floodwater pH. Under simulated flooded conditions, urea amended with Limus® reduced NH_3 loss by 36.6%, compared to urea alone, but floodwater pH had a significant effect on inhibitor efficacy. Key driving factors were air temperature, N application rate, and floodwater pH. The effectiveness and limitations of the inhibitor in NH_3 emission mitigation was examined, as well as its basis as one means of N pollution control in paddy rice cropping systems.

Keywords: NH_3 loss potential; rice field; floodwater pH; urease inhibitor

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1. Introduction

Rice production in China accounts for 30% of global rice production. Increasing applications of nitrogen (N) fertilizer fueled this, resulting in increased yields, but also large losses of N to the environment [1,2]. As one of the major pathways of N loss from rice, ammonia (NH_3) volatilization causes atmospheric pollution directly and by accelerating the formation of secondary inorganic aerosols, threatening visibility and public health [3]. Emitted NH_3 can also be transported and deposited to terrestrial ecosystems and water bodies, resulting in greenhouse gas emissions, soil acidification, eutrophication, and biodiversity loss [4–6].

The N fertilizer applied to rice paddy fields generally exceeds 300 kg N ha^{-1} yr^{-1}, with very low N use efficiency and substantial N losses [1,7,8]. Wang et al. suggested that N loss through NH_3 volatilization from paddy rice was several times higher than that through leaching or runoff [1]. The mechanism of NH_3 volatilization from paddy fields differs from that of upland agroecosystems. Therefore, understanding the potentially different controlling factors is essential for establishing mitigation strategies. The NH_3 volatilization rate (AVR), the proportion of N applied lost as NH_3, from rice, has been estimated by data-driven methods, but a large variation in the AVR was observed [9–11], which may be due to differences in the sample size and data selection criteria [2,9,12]. Recent research has suggested that taking account of the environmental conditions and farm management practices could reduce the uncertainty in estimating NH_3 emissions [13]. The relationships of NH_3 volatilization with soil type, climate, and N application rate have been studied, but some important factors, such as N management and meteorological conditions during the cropping season, as well as their variation in field experiments, have rarely been considered. This will impact the accuracy of the prediction of AVRs.

In attempts to reduce NH_3 losses, adding or combining (when formulating the fertilizer) a urease inhibitor (UI) with urea N fertilizer has been found to effectively retard the urease activity and, thus, the rate of hydrolysis of urea, consequently reducing NH_3 losses and improving agronomic performance [14,15]. Such inhibitors include urea analogues, such as phosphamide derivatives, that compete with urea to bind with soil urease. Those that efficiently inhibit urea hydrolysis include N-(n-butyl) thiophosphoric triamide (NBPT), N-(n-propyl) thiophosphoric triamide (NPPT), phenylphosphorodiamidate (PPD), and N-(2-nitrophenyl) phosphoric triamide (2-NPT). However, a previous meta-analysis has found that these products are less effective when applied to paddy rice, which may be caused by the dilution of the active ingredient by the floodwater or the slow formation of the active ingredient under anaerobic conditions [16]. The UI Limus® comprises 75% NBPT plus 25% NPPT and has been found to be effective across a range of soils, as well as very effective at reducing NH_3 emissions in dryland agroecosystems. However, the efficacy of Limus® for paddy rice has not been tested, and the factors influencing its efficiency remain unclear.

Our objectives, therefore, were to: (1) synthesize data from published literature reporting NH_3 volatilization from paddy rice in China and its correlations with a range of environmental and field management factors; (2) conduct an incubation experiment using 50 typical paddy soils sampled from the main rice producing areas across China and measure the NH_3 loss potential. NH_3 loss mitigation using the Limus® UI was investigated in the incubation experiment, and the influence of edaphic and floodwater properties were analyzed using a structural equation model. The overall aims were to obtain a robust estimation of the AVR in paddy rice, identify the key controlling factors, evaluate the efficacy of Limus® in paddy rice production, and improve our understanding of NH_3 loss from paddy rice and its mitigation options.

2. Materials and Methods

2.1. Data Synthesis

2.1.1. Data Collection

Peer-reviewed articles relating NH_3 volatilization from rice paddy in China, published between 1 January 1980 and 31 August 2021, were searched using key words 'NH_3 (or ammonia) volatilization' or 'NH_3 (or ammonia) emission' and 'paddy' or 'rice' in the Web of Science (WoS) and China Knowledge Resource Integrated (CNKI) databases. The collected articles were preliminarily screened to remove duplicate data, based on geographical information and experimental details. Retained papers were reviewed again, according to the following criteria: (1) only field and in situ lysimeter or pot experiments conducted in China were included; (2) experiments must include details of N applications (amount applied and the application method), include a corresponding control (without N addition), and the treatment and control must have been subjected to the same field management practices, such as irrigation, tillage, and P and K fertilizer applications; (3) the cumulative NH_3 losses from the experimental N treatment and the control were both recorded.

A total of 127 papers met the criteria, resulting in 700 paired observations. Cumulative NH_3 loss, weather conditions during crop growth (mean air temperature and rainfall), soil properties (pH, organic matter, total N content), and field management practices (N fertilizer type, N application rate, any split applications, and timings) were extracted. The NH_3 loss potential from paddy rice—the proportion of NH_3-N lost after subtracting background NH_3 emissions—as a proportion of total fertilizer-N application, was represented as the

NH_3 volatilization rate (AVR). The general mean AVR and corresponding 95% confidence interval (CI) for paddy rice was obtained by bootstrapping with 9999 iterations. Rice production systems were separated into single cropping, paddy-upland rotations, early rice (in double-cropping systems) and late rice (in double-cropping systems), based on cultivation regimes. AVRs in each system were evaluated using a non-parameter test (Wilcoxon), due to the uneven sample size and non-normal distribution of data. Statistical analyses were performed in R 4.2.1, in which bootstrapping and the Wilcoxon test were conducted using the 'boot' and 'ggsignif' packages, respectively.

2.1.2. Analysis of Influencing Factors of NH_3 Loss from Rice

In analyzing the factors controlling AVR in rice production, a linear model may not be able to describe the relationship between AVR and each explanatory variable, due to the random effects among the different studies. A linear mixed effect model was, therefore, adopted, in which the explanatory variables were considered as fixed effects, with each separate study as a random factor. Model performance was evaluated using ANOVA and the r.squaredGLMM test. The linear mixed effect model was established in R 4.2.1 using the 'lme4', 'car', and 'MuMIn' packages [17]. Differences between explanatory variables were evaluated by using a Wilcox test.

2.2. Soil Incubation

2.2.1. Soil Sampling and Pre-Incubation

Fifty typical paddy soils (topsoil, 0–20 cm) were collected from rice fields distributed across all of the rice-growing regions of China (detailed locations and soil properties are in Table S1). The soil samples were air-dried and ground to pass through 2 mm sieve. Soil pH, total N, C/N ratio, sand, silt, and clay contents were measured. A 100 g soil sample was placed in an incubation jar (8 cm diameter and 10 cm height), with 100 mL deionized water added (to simulate flooded field conditions), and the jars were sealed with plastic film pricked with several needle holes to restrict water (vapor) loss, while maintaining air exchange. The jars were put into a climate-controlled chamber at a constant temperature of 25 ± 0.2 °C and incubated for two weeks. After incubation, jars were taken out, any lost water replaced, and prepared for fertilizer addition and NH_3 measurement.

2.2.2. Fertilizer Application

The incubation experiment (without rice) comprised two fertilizer treatments: 1) U, urea powder (99.99%, manufactured by Sinopharm Chemical Reagent Co. Ltd., Shanghai, China), 2) U+UI, urea powder amended with urease inhibitor (Limus®; 75% NBPT and 25% NPPT; supplied by BASF SE, Ludwigshafen, Germany). The inhibitor was added to the urea and mixed by shaking. The UI dose was 0.5% of urea by weight. A total of 110 mg of each fertilizer was added to each pre-incubated jar, and all jars sealed with a specially made lid with a hole in the middle, fitted with a rubber bung. Each treatment was replicated three times.

2.2.3. NH_3 Measurement

NH_3 loss after fertilizer application was measured using Dräger-Tubes (supplied by Drägerwerk AG & Co. KGaA, Lübeck, Germany; tube type 'Ammonia 20/a-D'), as detailed in Sha et al. (2020) [18]. Each tube contained acid to absorb NH_3 and bromophenol blue and to reflect acid absorption as a change of color. A tube was insert into the lid of each jar and NH_3 concentration monitored using a color index. When the index reached a maximum (equivalent to 1500 ppm), a new tube was exchanged. The cumulative NH_3 concentration over time (one week) was determined and presented as the NH_3 lost from each soil.

2.2.4. Analysis of Factors Influencing NH_3 Loss

The initial floodwater pH (IFW pH) before the fertilizer was added and the floodwater pH at the end of experiment were measured, and the percentage change between the two measurements 'ΔFW pH' calculated. The IFW pH, ΔFW pH, soil pH, total N, C/N ratio, sand, silt, and clay contents were selected as explanatory variables. The NH_3 loss and reduction in loss caused by using the inhibitor were treated as response variables. A structural equation model (SEM) was used to interpret the correlation of explanatory variables with response variables. A knowledge-based conceptual model of hypothetical relationships was first established, the Satorra-Bentler corrected maximum likelihood χ^2 statistic was used to assess model performance, for which a p value above 0.05 is acceptable [19]. Any non-significant path was systematically removed, and the resulting revised model, compared with the previous one, using the Akaike information criteria (AIC) value, in order to evaluate the suitability of path deletion. The final model gave the standardized coefficients of each path, as well as the R^2 of each explanatory variable. SEM model analysis was conducted in R 4.2.1, using the 'lavaan' package.

3. Results

3.1. NH_3 Loss from Rice Production in China

The data synthesis showed the mean NH_3 loss (AVR) from all rice production systems in China to be 16.2% (Figure 1A). The AVRs of rice in single cropping, paddy-upland rotations, early rice, and late rice in double cropping were 13.0%, 14.2%, 19.4%, and 22.1%, respectively (Figure 1B). Those of early rice and late rice were significantly higher than the others, but no significant difference was found between the early and late rice. The AVR from surface-applied N was significantly higher than that from subsurface applied N (Figure 2A). Among different N fertilizer types, ammonium bicarbonate resulted in the highest AVR, followed by urea (also higher than others except for compounds), and the lowest AVR was from organic fertilizer (Figure 2I). Temperature during the rice growing season and the N application rate positively correlated with AVR (Figure 2C,E), but the basal N ratio, soil organic matter, and total N content were negatively correlated with AVR (Figure 2B, F and H).

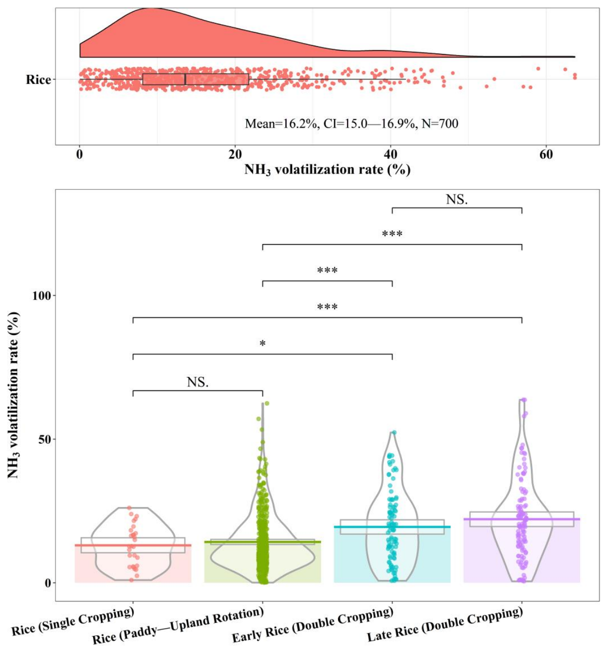

Figure 1. NH_3 loss from different rice production systems in China: (**A**) general mean AVR in rice fields from across China: CI is the confidence interval generated by bootstrapping; N is the number of observations; (**B**) mean AVR of different rice-based production systems: an asterisk (*) between two columns implies a significant difference, with * and *** indicating significance levels at 0.05 and 0.001, respectively. NS between two columns implies a non-significant difference.

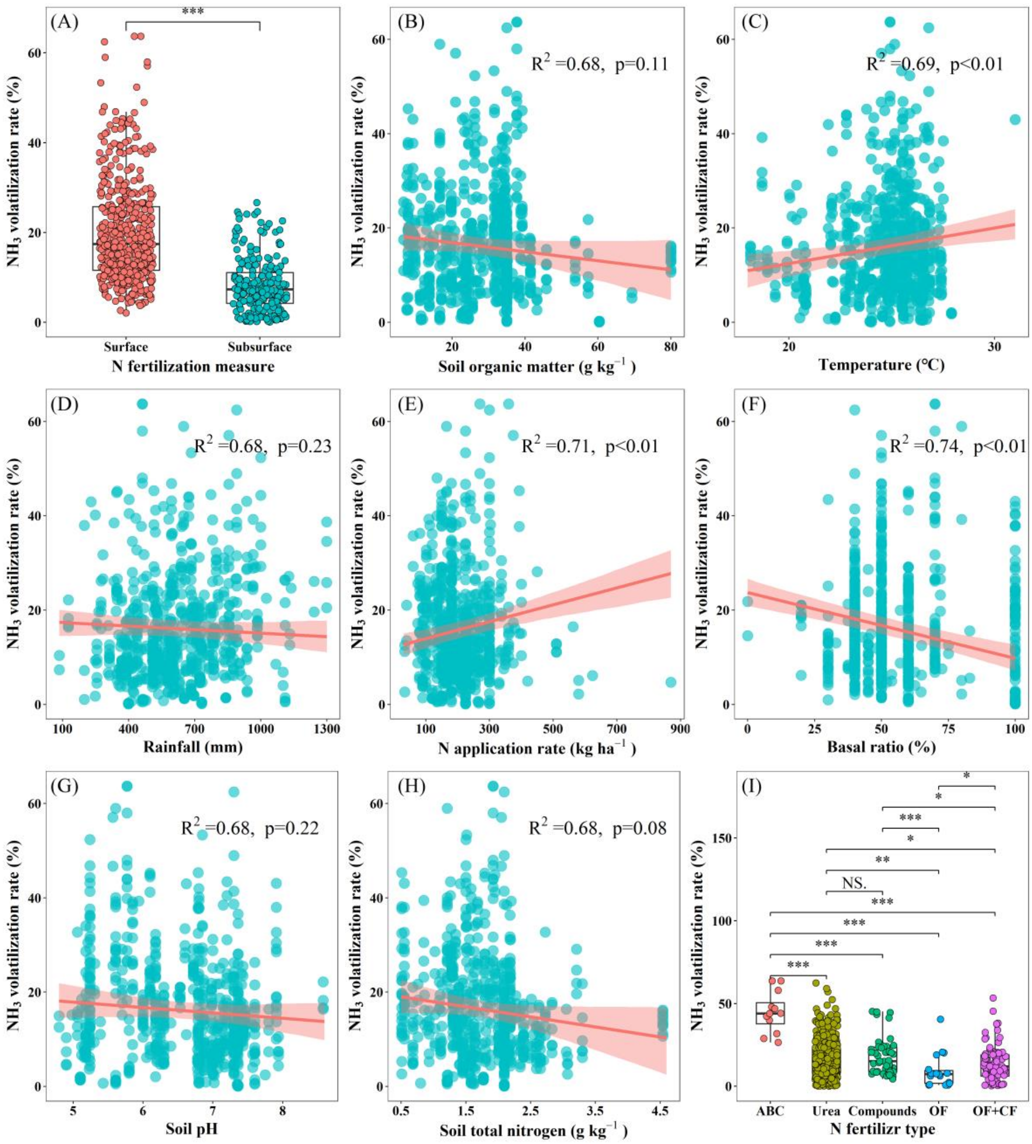

Figure 2. Influencing factors of NH_3 losses from rice production systems across China. The asterisks (*) in (**A**) and (**I**) show significant differences between groups, with *, **, and *** indicating significance levels at 0.05, 0.01, and 0.001, respectively. The solid line and surrounding shaded areas in (**B**–**H**) represent the predicted value and 95% confidence interval of the linear mixed effect model, respectively.

3.2. NH_3 Loss and Reduction Potential in Paddy Soils

According to the results from the incubation experiment, paddy soils from Northeast China generally had low NH_3 losses (Figure 3A and Figure S1A); the soil with the highest NH_3 loss was found in South China (Figure S1C), followed by Central China (Figure S1D), East China (Figure S1B), and Southwest China (Figure S1E). Compared to the urea treatment (U), the addition of the inhibitor (UI) significantly reduced NH_3 losses across all paddy soils by 36.6% (Figure 3B and Figure S2). The SEM model suggested that the NH_3 loss was significantly affected by soil pH, sand content, IFW pH, and ΔFW pH (Figure 4). After urea application, floodwater pH increased by 11.7%, but adding the inhibitor significantly reduced this pH increase (Figure S2). The NH_3 loss reduction potential of the inhibitor was significantly influenced by soil pH, SOC, and IFW pH. A negative correlation was found between NH_3 loss and NH_3 reduction potential.

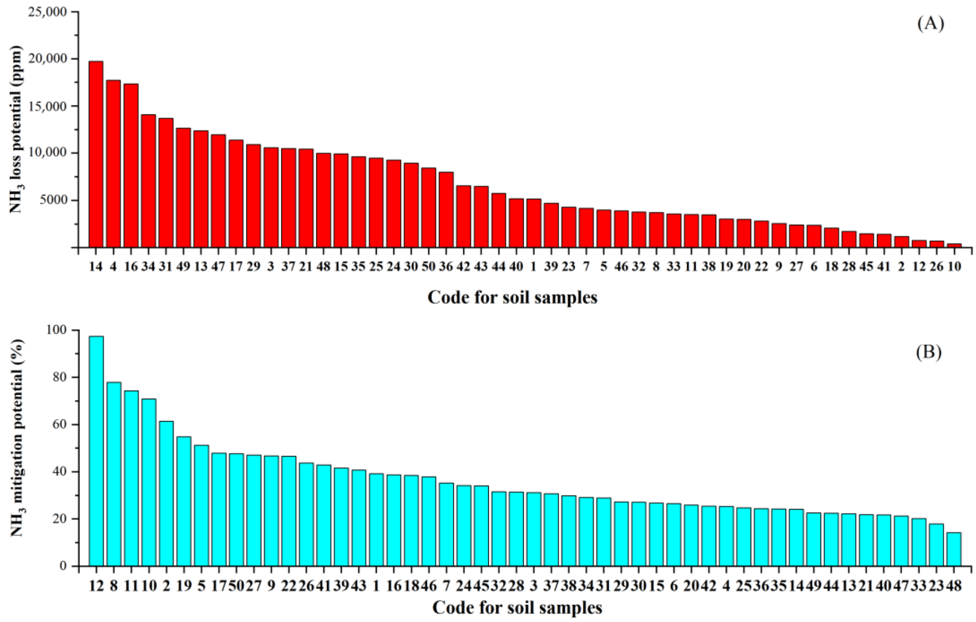

Figure 3. Factors influencing NH_3 losses from rice systems across China. (**A**) NH_3 loss from of 50 paddy soils from across China, (**B**) NH_3 reduction efficiency of using urea fertilizer with a urease inhibitor (Limus®, the UI treatment) compared to urea. The number along the X axis is the code number for each paddy soil, as detailed in Table S1.

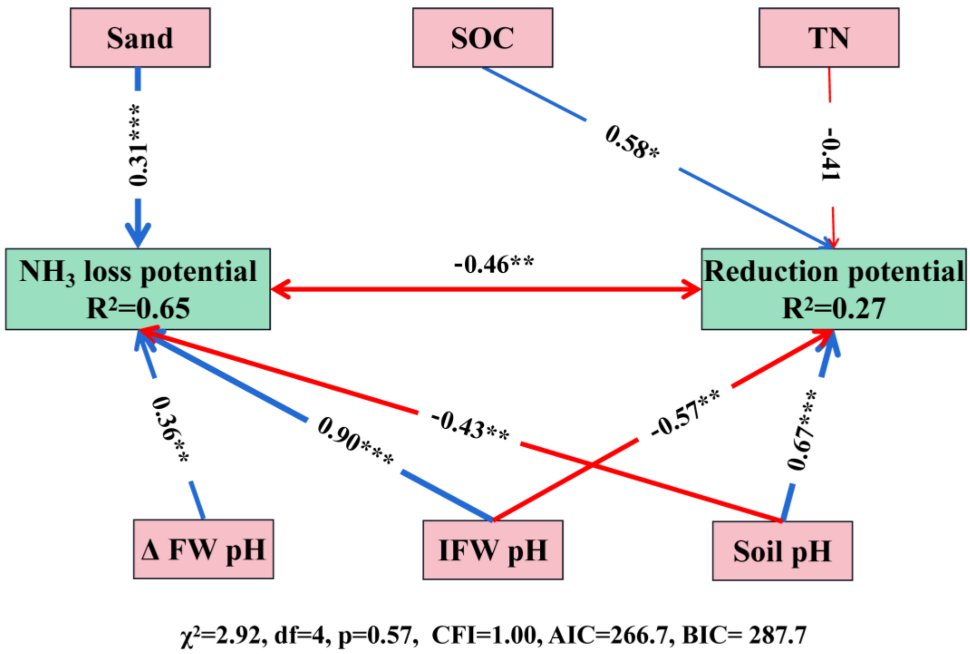

Figure 4. The structural equation model analysis of factors influencing NH_3 loss and the reduction potential of using the urease inhibitor. The NH_3 loss from 50 typical paddy soils under simulated flooded condition and the corresponding NH_3 reduction potential by using the Limus® urease inhibitor (UI) were considered as the response variables. Soil and flooded water properties were explanatory variables. Blue and red arrows indicate positive and negative relationships, respectively. Numbers on the arrows indicated the standardized path coefficients, and asterisks show significant impacts of explanatory variable on the corresponding response variable, with *, **, and *** indicating significance levels at 0.05, 0.01, and 0.001, respectively. The thickness of the arrows indicates the magnitude of the correlation.

4. Discussion

4.1. NH_3 Loss from Paddy Rice in China and Controlling Factors

The NH_3 loss from paddy rice in China has been evaluated before using data-driven methods. For example, Chen et al. (2014) estimated the AVR of rice in China to be 16.0% using a linear model across 265 sites [10]. Ma et al. (2020) summarized 358 field NH_3 measurements, finding that 13.8% of the N applied to rice were lost through NH_3 volatilization [9]. Zhou et al. (2016) found a mean AVR of 15.0% [11]. Our estimation was derived from a dataset of 700 observations, and the mean AVR (16.2%) was slightly higher than previous estimates. Clearly, NH_3 losses from different rice-based cropping systems vary greatly. Double rice cropping systems have the highest AVR, as found by Wang et al. (2018) [1]. Jiang et al. (2016) suggested that bacterial and fungal diversity were lower in double rice systems than in rice-wheat rotations [21], which may impact the abundance of ammonia oxidizer and consumption of ammonia. Continuously waterlogged conditions may lead to a low redox potential, which slows N transformations, such as nitrification, prolonging the residence time of the substrate of NH_3 loss in paddy fields [1,22] Single cropped rice is mainly grown in high latitude areas with low annual mean temperatures and high organic matter, which can cause low AVRs, consistent with the findings of our incubation experiment, which found relatively low NH_3 losses from paddy soils from Northeast China.

Unlike NH_3 loss from upland soils, NH_3 volatilization from paddy rice occurs at the floodwater–atmosphere interface. The properties of the floodwater, such as NH_4^+-N content, pH, and temperature, strongly affect the NH_4^+-NH_3 equilibrium. Unfortunately, our data synthesis did not include an analysis of all floodwater properties, due to a lack of available data. However, the results from the incubation experiment suggested that IFW pH and ΔFW pH were positively correlated with NH_3 loss. An increase in floodwater pH after urea application was observed in all the soils, resulting in the rapid dissociation of NH_4^+-N, leading to substantial NH_3 accumulation and loss [23]. Additionally, proton release during the oxidation of NH_4^+ to NO_3^- is impeded by the low Eh in flooded soil, which can further stimulate NH_3 loss from paddy soils [15]. H^+ in soil acts as a buffer to changes in soil pH, providing acid soils a relatively low NH_3 loss potential [24]. This was not the case in our incubation experiment. The growth of algae in floodwater could increase floodwater pH during the photosynthetic uptake of CO_2 [25,26]. Thus, floodwater pH, rather than soil pH, appears to control NH_3 loss under flooded conditions, explaining most non-significance of soil properties in the data synthesis. However, the negative correlation between NH_3 loss and soil pH merits further analysis, including more soils with a wider range of pH values.

Types, amounts, and timings of fertilizer application were also significant factors impacting NH_3 loss from flooded soils. The IPCC Tier 1 emission calculations assume that NH_3 loss (as kg N ha^{-1}) responds linearly to the N application rate (NAR), although non-linear relationships have been widely reported [11]. Using the AVR, instead of actual N loss, the linear mixed effect model suggested a positive correlation with NAR. Applying N below the surface of the soil generally prevented NH_4^+ diffusion to the floodwater surface and, therefore, reduced NH_3 volatilization and the AVR, and so, the enhanced crop uptake [27]. Mixing the N fertilizer with surface soil is common practice for basal N applications to rice, which acts similar to a subsurface application, explaining the negative relationship of basal N with AVR. The low AVR associated with organic forms of N can be attributed to the slow mineralization rate, and so, the limited accumulation of NH_3 in floodwater. In contrast, ammonium bicarbonate and urea use result in substantial NH_4-N accumulation and an increase in floodwater pH, explaining the high NH_3 loss [24].

4.2. Reduction in NH_3 Losses Achieved with a Urease Inhibitor and Influencing Factors

Using Limus® retarded urea hydrolysis and significantly reduced NH_3 loss after fertilizer application, a previous meta-analysis suggested that using urea with an inhibitor could reduce NH_3 loss (compared to plain urea) across all crop types, on average by

64.6%, but the reduction in NH_3 losses from rice only reached 42.1%, significantly less [15]. The low efficacy of the inhibitor in rice could be due to the inhibition of hydrolysis of NBPT (converted to NBPTO, the actual ingredient for inhibiting urease) under anaerobic conditions or the dilution of the active ingredient by floodwater [16].

The SEM model suggests that the reduction potential of the inhibitor was negatively related to NH_3 loss and IFW pH, when the soils used in the incubation study were pre-incubated for two weeks under flooded conditions. Interestingly the soil with the highest IFW pH also had noticeable algal growth in the floodwater (data not shown). The biodegradation of the inhibitor by the algae could explain the low efficacy in the high IFW pH soil.

5. Conclusions

The subsurface application of urea, using urea with a urease inhibitor, such as Limus®, or using an organic form of N can reduce the AVR in paddy rice. However, the dose of Limus® in our incubation study is almost ten times that used in upland soils, which may not be economical for use on farms and may also raise environmental or food safety issues, e.g., exceeding the maximum dose in EU regulations [20]. Floodwater pH was a key factor regulating NH_3 loss from paddy soils and the efficacy of the inhibitor. Optimizing the N application rate, combined with a urease inhibitor, offers one solution for reducing N losses from rice-based cropping systems, providing realistic options for sustainable N management and green agricultural development. Future developments of inhibitors, such as Limus®, for rice should consider dose rates, plus relationships with water table height, e.g., lowering the water table, and their combined application with an algicide.

Supplementary Materials: The following supporting information can be downloaded at: https://www.mdpi.com/article/10.3390/atmos13111750/s1, Table S1: Information of soils used in incubation experiment; Table S2: List of literature used in the data synthesis. Figure S1: Dynamics of NH_3 loss after fertilizer application to 50 flooded paddy soils in China. Figure S2: Differences in NH_3 loss potential and changes in flooding water pH under the two treatments: urea (U) and urea plus the Limus®inhibitor (U_UI). Asterisks show significant differences between treatments, with *, **, and *** indicating significance levels at 0.05, 0.01, and 0.001, respectively.

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