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Effect of Nitrogen Fertilizer Type and Application Method on Ammonia Volatilization from a Calcareous Soil in Nineveh Governorate, Iraq

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ABSTRACT

Despite the critical role of nitrogen in enhancing crop productivity, its efficiency remains significantly low in the calcareous soils of northern Iraq due to rapid ammonia (NH_3) volatilization. Therefore, this comprehensive study was conducted to evaluate the effects of different nitrogen fertilizer types (urea, ammonium sulfate, and ammonium chloride) and application methods (surface vs. subsurface at 3 cm depth) on ammonia volatilization from a calcareous soil in Nineveh Governorate, Iraq, under local temperature conditions ranging from 0 to 23 °C. The integration of these chemical and physical factors aims to provide a clearer understanding of nitrogen dynamics in semi-arid environments and to identify the most sustainable and efficient management practices for local agricultural systems. The study followed a completely randomized design (CRD) with three replicates ($n = 3$). Ammonia loss was monitored over a 20-day incubation period, with measurements taken at four intervals at 5-day intervals (days 5, 10, 15, and 20). The results demonstrated that subsurface placement significantly mitigated nitrogen loss, reducing cumulative ammonia volatilization by an average of 22.83% compared to surface application. Among the nitrogen sources, ammonium sulfate (F3) exhibited the highest cumulative volatilization (14.09 mg pot^{-1}), while urea (F2) showed the lowest volatilization (12.28 mg pot^{-1}) under the experimental conditions. These findings emphasize that integrated fertilizer management strategies are essential for improving nitrogen use efficiency and reducing environmental losses in the calcareous soils of Northern Iraq.

1. INTRODUCTION

Nitrogen (N) is considered one of the most essential nutrients required by plants due to its significant role in many physiological functions. It is involved in the synthesis of amino acids and is a key component of chlorophyll, which gives it importance in the photosynthesis process. In addition, nitrogen is vital for respiration and provides the energy required by the plant to complete its metabolic activities. It is also involved in the formation of protoplasm, enzyme cofactors such as $NADPH_2$ and $NADH_2$, as well as vitamins like vitamin B and vitamin H, and several plant growth regulators such as auxins, cytokines, and alkaloids [1-3]. Most soils suffer from a deficiency in the available nitrogen content, which necessitates its addition in the form of fertilizers to compensate for this shortfall and increase its availability. However, the applied nitrogen is subject to various loss pathways that negatively affect plant growth and productivity. One of the most significant of these is nitrogen loss through ammonia volatilization [4, 5]. This process is primarily driven by high soil pH and calcium carbonate ($CaCO_3$) content, which facilitates the chemical equilibrium shift toward ammonia gas formation. This occurs particularly when ammonium nitrogen fertilizers are surface-applied to alkaline soils. In such conditions, ammonium is converted to ammonia gas through the following reaction:

$$NH_4^+ + H_2O + OH^- \rightarrow NH_3 + 2H_2O$$

Ammonia volatilization from nitrogen fertilizers, especially in calcareous soils, is one of the major challenges that reduces fertilizer efficiency [6]. From an economic perspective, this loss represents a significant reduction in fertilizer value, in addition to contributing to environmental pollution via gaseous emissions. The amount of nitrogen lost through this process ranges between 9–64% of the applied nitrogen, with an average of approximately 14% [7]. The extent of nitrogen loss as ammonia depends on several factors, including soil texture, moisture, calcium carbonate content, air temperature, fertilizer type, method, and rate of application, and fertilizer decomposition speed [8, 9].

The type of nitrogen fertilizer affects the amount of ammonia volatilized due to differences in its chemical composition and physical properties. Studies have shown that volatilization rates vary among nitrogen fertilizers, even when applied at equal nitrogen rates [10]. For example, the study [11] reported that the cumulative volatilization loss over 18 weeks followed the order: $(NH_4)_2SO_4 > CO(NH_2)_2 > NH_4NO_3$.

The study [12] also noted higher ammonia losses from ammonium sulfate than from urea. The study [13] found volatilization losses of 4.9% for ammonium sulfate and 7.9% for zinc-coated urea.

The method of fertilizer application is another critical factor influencing nitrogen use efficiency. Using the appropriate application technique can help overcome unfavorable conditions that limit nutrient availability. It has been observed that surface application results in the highest ammonia losses [14-16], while even a 1 cm soil cover can significantly reduce volatilization, and deep placement nearly eliminates it. The study [17] reported a 65% nitrogen recovery by plants when urea was applied at an 8 cm depth compared to only 39% recovery when surface-applied. Placing urea or organic fertilizer below the soil surface leads to a significant reduction in the amount and rate of ammonia volatilization compared with surface application, with losses decreasing by up to 50–80%, this reduction is attributed to decreased contact of the fertilizer with air and increased retention within the soil [18]. In dry farming systems, applying urea at a depth of 10 cm reduced NH_3 losses by about 62% compared to applying it on the surface [19]. Also, the study [20] indicated that deep fertilizer placement can effectively suppress ammonia volatilization and reduce nitrogen losses, noting that applying nitrogen fertilizers at a 10 cm depth increased nitrogen recovery efficiency by 18.1–52.3% compared to surface application.

There is a critical need to focus on the specific volatilization mechanisms and the regional context of calcareous soils in Northern Iraq. In Nineveh Governorate, despite the prevalence of highly calcareous soils (exceeding 25% $CaCO_3$), there is a notable lack of quantitative research dealing with this issueThe highest amount of ammonia volatilization from nitrogen fertilizers occurs shortly after addition, especially during the first days, and then the amount gradually decreases thereafter [21], As noted by the study [22] that the evaporation of ammonia from the soil occurs mainly during the first seven days after fertilization, and then the differences between the different fertilization methods decrease little by little. Due to the large amount of nitrogen lost through volatilization and the lack of research dealing with this issue. This study was conducted to evaluate the efficiency of different types of nitrogen fertilizers and their placement in calcareous soil based on the amount of volatilized ammonia. The present study was specifically designed to investigate the complex dynamics of ammonia (NH_3) volatilization in the calcareous soils of Nineveh, Iraq. By examining four distinct nitrogen fertilizer types and two strategic application methods (surface and subsurface) under fluctuating local temperatures (0–23 ℃), this research aims to identify high-efficiency management practices that minimize nitrogen loss and support sustainable agriculture in semi-arid environments.

2. MATERIALS AND METHODS

2.1 Soil properties

A surface soil sample (0–30 cm) was collected from Tel Abta, Nineveh. Physicochemical analysis identified the soil as a silty clay loam. The initial properties were: pH 7.30, EC 2.30 dS m^{-1}, $CaCO_3$ content 29.5% (295 g kg^{-1}), and CEC 27.7 Cmol(+)/kg.

2.2 Experimental setup

Plastic pots (17 cm diameter, 7 cm depth) were filled with 1 kg of soil. The experiment was conducted in an outdoor rain shelter to reflect regional temperature dynamics (0–23 ℃). Four fertilizer treatments (F1-F4) were applied at 100 mg N kg^{-1} soil with three replicates (n = 3).

2.3 Application methods

1. D1 (Surface): Fertilizers were broadcast on the soil surface.
2. D2 (Subsurface): Fertilizers were applied at a depth of 3 cm and covered with a layer of soil to minimize direct air contact.

2.4 Ammonia volatilization measurement

Ammonia volatilization was captured and quantified using a specialized static closed-chamber system. A 50 mL glass beaker containing 20 mL of 2% boric acid (H_3BO_3) mixed with a bromocresol green-methyl red indicator was carefully positioned inside the chamber directly on the soil surface. To ensure the high precision of the measurements and to prevent any potential atmospheric gas exchange or external contamination, all chambers were strictly sealed using airtight rubber gaskets and reinforced industrial-grade adhesive sealing tape. This rigorous sealing protocol guaranteed that all captured ammonia originated exclusively from the treated soil area. The acid traps were replaced at each sampling interval to maintain continuous and maximum absorption capacity throughout the experimental period. Pots were sealed with non-reactive plastic covers to prevent gas leakage. Titration was performed every 5 days (5, 10, 15, 20 days) against 0.014 N HCl [23]. Table 1 shows the coordinates and classification of the soil.

The soil was air-dried and sieved through a 2 mm mesh. Table 2 shows some of the chemical and physical properties of the soil used in the study, which were determined according to the methods presented in studies [24, 25].

The experiment involved four types of nitrogen fertilizers: F1: control (without fertilizer), F2: Urea [$CO(NH_2)_2$] 46% N, F3: Ammonium sulfate [$(NH_4)_2SO_4$] 21% N, F4: Ammonium chloride [NH_4Cl] 26% N. Each fertilizer was applied at a single nitrogen level of 100 ppm N, using plastic pots with a diameter of 17 cm and a depth of 7 cm, filled with 1 kg of dry soil.

Fertilizers were applied using two methods: D1: surface application: fertilizer spread on the soil. D2: subsurface application: fertilizer placed beneath the topsoil layer. A small glass beaker containing 20 mL of boric acid solution (2% boric acid + mixed indicator) was placed on the soil surface in each pot to trap the volatilized ammonia during measurement intervals. Pots were watered to 75% of field capacity and immediately sealed tightly to prevent ammonia gas escape. The experiment was conducted with three replicates. Ammonia volatilization was measured every five days over four periods P1, P2, P3, P4 (five days period). Detection was based on the color change of the boric acid solution from purple to green, followed by titration with 0.014 N hydrochloric acid according to the study [26] and the procedure applied by the study [6]. Each time a boric acid trap was removed for analysis, it was replaced with a fresh one.

Soil moisture was maintained at 75% field capacity throughout the experiment by weighing the pots and replenishing lost water.

Table 1. Location, classification, and agricultural use of the experimental soil

Property	Value
Location	Tal Abta Subdistrict/Nineveh Governorate
Longitude	42.4442 °E
Latitude	35.6942 °N
Soil order	Aridisols
Suborder	Calcids
Great group	Haplocalcids
Agricultural utilization	Cereal cultivation (wheat and barley)

Table 2. Some physical and chemical properties of the experimental soil

Property	Value	Unit
Soil separates		
pH (1:1 soil: water)	7.30	
EC	2.30	dS m^{-1}
Organic matter	12.30	g kg^{-1}
Total calcium carbonate	295.00	
CEC	27.70	cmol(+)/kg
Available N	34.00	mg kg^{-1}
Available P	1.15	
Available K	192.00	
Field capacity	190.00	g kg^{-1}
Bulk density	1.30	Mg m^{-3}
Sand	189.00	g kg^{-1}
Silt	525.00	
Clay	286.00	
Soil texture	Silty clay loam	

The experimental data were subjected to a rigorous three-way Analysis of Variance (ANOVA) using the GenStat software package. The study followed a 4 × 2 × 4 factorial arrangement involving four fertilizer types (F), two application methods (D), and four sampling periods (P) in a completely randomized design (CRD). Since measurements were collected from the same experimental units (pots) over multiple time intervals, the 'Period (P)' factor was treated as a repeated-measures factor to account for temporal correlations. Before conducting the ANOVA, fundamental assumptions, including the normality of distribution and homogeneity of variance, were verified using Levene's test. Mean separations were performed using the Least Significant Difference (LSD) test at a significance level of $p < 0.05$. Significance annotations (lowercase letters) were integrated into all tables and figures to facilitate the interpretation of treatment effects.

3. RESULTS AND DISCUSSION

3.1 Effect of nitrogen fertilizer type and application method on ammonia volatilization

Table 3 and Figure 1 show the effect of different nitrogen fertilizer types and application methods on the amount of ammonia volatilized (measured in mg pot^{-1}). It was found that nitrogen loss as ammonia occurred in all fertilized treatments, and it was significantly superior to the control treatment, which recorded the lowest volatilization amount (0.15 mg pot^{-1}).

The chemical and physical properties of the experimental soil are presented in Table 2. It can be observed that the soil has a basic pH (7.30), high calcium carbonate content (295 g kg^{-1}), and low organic matter content (12.30 g kg^{-1}); these characteristics contributed to a low concentration of available phosphorus (1.15 mg kg^{-1}). The soil texture was classified as silty clay loam, with a high silt content and low sand content. Due to these properties, especially the high soil pH resulting from elevated calcium carbonate, the nitrogen applied in the form of ammonium fertilizers is prone to loss as ammonia gas via volatilization [27, 28]. This loss reduces fertilizer efficiency and contributes to the negative effects of ammonia gas, such as inhibition of seed germination and root scorching [29, 30].

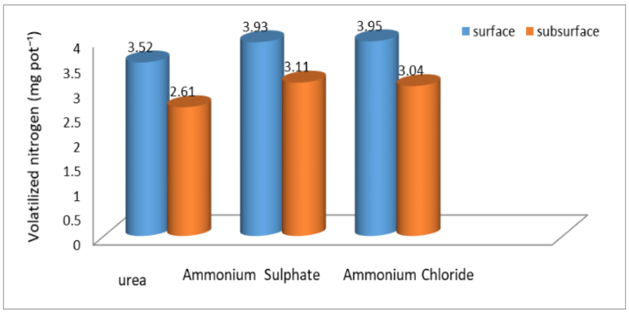

Figure 1. Effect of nitrogen fertilizer type and its placement on the amount of volatile nitrogen (mg pot^{-1}) during the experiment

The highest ammonia loss occurred with ammonium sulfate (3.52 mg pot^{-1}), while the lowest was from urea (3.07 mg pot^{-1}), these differences may be due to the chemical composition and physical properties of the fertilizers, the lower ammonia volatilization from urea might also be attributed to slower urea hydrolysis due to low temperatures during the experiment period (Table 4) which reduced microbial activity and delayed the conversion of urea to ammonium, thereby decreasing volatilization, these findings align with those of studies [13, 31, 32].

The method of fertilizer placement significantly affected nitrogen volatilization; the surface application recorded the highest average loss (2.89 mg pot^{-1}), while subsurface application reduced this to 2.23 mg pot^{-1}, representing a 22.83% reduction. Subsurface placement prevents contact between fertilizer and the external environment, limiting exposure to air, temperature, and wind, which all promote volatilization. It also facilitates direct interaction with soil particles rich in protons (H^+), organic matter, and clay minerals that stabilize ammonia by converting it to ammonium (NH_4^+). These results agree with studies [20, 31, 33], which observed reduced volatilization with deep fertilizer placement.

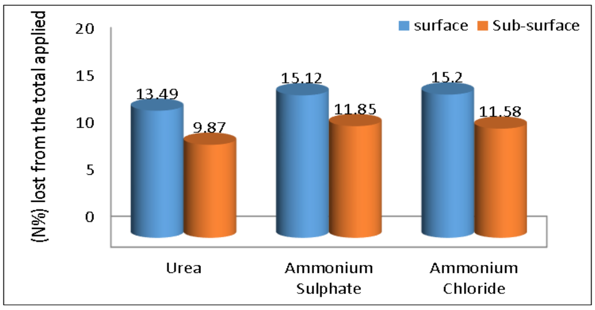

Figure 2. Effect of nitrogen fertilizer type and its placement on N% lost from the total applied nitrogen

3.2 Effect of fertilizer type and application method on cumulative ammonia volatilization

Table 5 shows the cumulative ammonia loss across all treatments. The control (no fertilizer) recorded the lowest loss (0.60 mg pot^{-1}), while the fertilizers ranked in the following order: $(NH_4)_2SO_4$ > NH_4Cl > $CO(NH_2)_2$, with ammonium sulfate leading at 14.08 mg pot^{-1}, followed by ammonium chloride (13.99 mg), and urea (12.28 mg). The percentage of nitrogen lost via volatilization for surface application was: 15.12%, 15.20%, and 13.49%, for urea, ammonium sulfate, and ammonium chloride, respectively. For subsurface application, the values dropped to 11.85%, 11.58%, and 9.87%, respectively (Figure 2). These results are consistent with those reported by the study [13].

Table 3. Effect of nitrogen fertilizer type and method of adding it on the amount of volatile ammonia (mg pot^{-1}) during the different measurement periods

Fertilizer (F)	Addition Method	Measurement Period (P)				D × F	(F) Effect	(D) Effect
		P1	P2	P3	P4			
F1	D1	0.17	0.15	0.15	0.13	0.15	0.15	
	D2	0.17	0.15	0.15	0.13	0.15		
F2	D1	4.24	3.90	3.10	2.85	3.52	3.07	D1 2.89
	D2	3.12	2.85	2.30	2.20	2.61		D2 2.23
F3	D1	5.17	3.90	3.60	3.05	3.93	3.52	
	D2	4.30	3.10	2.80	2.25	3.11		
F4	D1	5.20	4.10	3.55	2.95	3.95	3.49	
	D2	3.90	3.15	2.71	2.42	3.04		
F × P	F1	0.17	0.15	0.15	0.13		LSD at 0.05 level	
	F2	3.68	3.37	2.70	2.52		D: 0.20	
	F3	4.73	3.50	3.20	2.65		F: 0.29	
	F4	4.55	3.62	3.13	2.68		P: 0.29	
	D1	3.69	3.01	2.60	2.24		D × F: 0.41	
	D2	2.87	2.31	1.99	1.75		D × P: 0.41	
(P) Effect		3.28	2.66	2.29	1.99		F × P: 0.58 F × D × P: 0.83	

Notes: Each value in the table represents the mean of three replicates ± 0.416; LSD: Least Significant Difference.

Table 4. Minimum and maximum temperatures (°C) during the experimental period

Period	Minimum Temperature	Maximum Temperature
16 /2/ 2025	0	15
17 /2/ 2025	3	15
18 /2/ 2025	4	14
19 /2/ 2025	3	12
20 /2/ 2025	6	13
21 /2/ 2025	5	12
22 /2/ 2025	1	11
23 /2/ 2025	0	10
24 /2/ 2025	0	10
25 /2/ 2025	0	9
26 /2/ 2025	1	11
27 /2/ 2025	1	13
28 /2/ 2025	1	12
1 /3/ 2025	1	15
2 /3/ 2025	2	15
3 /3/ 2025	4	19
4 /3/ 2025	4	20
5 /3/ 2025	6	21
6 /3/ 2025	8	23
7 /3/ 2025	8	22

Table 5. Effect of nitrogen fertilizer type and method of addition on the amount of accumulated volatile ammonia (mg pot^{-1})

Addition Method	Fertilizer				(D) Effect
	F1	F2	F3	F4	
D1	0.60	14.09	15.72	15.80	11.55
D2	0.60	10.48	12.45	12.18	8.93
Effect (F)	0.60	12.28	14.09	13.99	
LSD at 0.05 level	D: 0.69		F: 0.98	D × F: 1.39	

Notes: Each value in the table represents the mean of three replicates ± 0.66; LSD: Least Significant Difference.

3.3 Effect of fertilizer type and application method on ammonia volatilization rate

Table 6 indicates that the lowest ammonia volatilization rate was recorded with urea (0.614 mg NH_3 day^{-1}), whereas the highest rate was observed with ammonium sulfate (0.704 mg NH_3 day^{-1}), followed by ammonium chloride (0.699 mg NH_3 day^{-1}). Urea exhibited the slowest volatilization rate, which may be attributed to the specific physical and chemical properties of each fertilizer type. Urea must first be hydrolyzed to ammonium through enzymatic activity, a process that is strongly influenced by soil microbial activity and temperature. During the experimental period, temperatures were relatively low (0–23 ℃), which likely slowed urea hydrolysis and delayed ammonium production, thereby reducing NH_3 volatilization. In contrast, ammonium ions become immediately available following the application of ammonium sulfate and ammonium chloride, leading to a more rapid volatilization process.

Regarding the method of application, subsurface placement resulted in a 29.9% reduction in ammonia volatilization compared with surface application, the volatilization rate under surface application was 0.578 mg NH_3 day^{-1}, compared with 0.446 mg NH_3 day^{-1} for subsurface application, this reduction may be attributed to the fact that subsurface application minimizes fertilizer exposure to atmospheric conditions such as air movement, wind, and temperature, in addition, subsurface placement enhances the formation of ammonium ions through reactions with protons (H^+) and soil colloids, including clay minerals and organic matter, which increases ammonium retention in the NH_4^+ form and consequently reduces the rate of ammonia volatilization.

The time period had a pronounced effect on the rate of ammonia volatilization from the applied fertilizers, the highest volatilization rate was observed during the first period (P1 = 0.657 mg NH_3 day^{-1}), followed by a gradual decline in subsequent periods (P2 = 0.533, P3 = 0.459, and P4 = 0.399 mg NH_3 day^{-1}), this trend can be attributed to the rapid fertilizer hydrolysis immediately after application and the formation of unstable ammonium carbonate, which decomposes quickly into CO_2 and NH_3, as the fertilizer is progressively consumed, the volatilization rate decreases. Therefore, special attention should be given to fertilizer management during the first few days after application to minimize nitrogen losses. These results are consistent with studies [34-36], which also reported that the highest volatilization occurs shortly after fertilizer application.

Table 6. Effect of nitrogen fertilizer type and application method on the rate of ammonia volatilization (mg NH_3 day^{-1})

Fertilizer (F)	Addition Method	Measurement Period (P)				D × F	(F) Effect	(D) Effect	
		P1	P2	P3	P4				
F1	D1	0.034	0.030	0.030	0.026	0.030	0.030		
	D2	0.034	0.030	0.030	0.026	0.030			
F2	D1	0.848	0.780	0.620	0.570	0.705	0.614	D1	0.578
	D2	0.625	0.571	0.460	0.440	0.524		D2	0.446
F3	D1	1.034	0.780	0.720	0.610	0.786	0.704		
	D2	0.860	0.620	0.560	0.450	0.623			
F4	D1	1.020	0.820	0.710	0.590	0.790	0.699		
	D2	0.780	0.630	0.542	0.484	0.609			
F × P	F1	0.034	0.030	0.030	0.026		LSD at 0.05 level		
	F2	0.736	0.675	0.540	0.505		D: 0.20		
	F3	0.947	0.700	0.640	0.530		F: 0.29		
	F4	0.910	0.725	0.626	0.537		P: 0.29		
	D1	0.739	0.603	0.520	0.449		D × F: 0.41		
	D2	0.575	0.463	0.398	0.350		D × P: 0.41		
(P) Effect		0.657	0.533	0.459	0.399		F × P: 0.58 F × D × P: 0.83		

Notes: Each value in the table represents the mean of three replicates ± 0.083; LSD: Least Significant Difference.

The dynamics of NH_3 loss during the trial were profoundly influenced by the ambient temperature fluctuations (0–23 °C) recorded in Nineveh. A notable observation was the delayed peak of volatilization in urea treatments (F2) compared to ammonium-based fertilizers. This phenomenon is scientifically attributed to the inhibitory effect of low temperatures on urease enzyme activity. According to the Arrhenius principle, enzymatic reactions are highly sensitive to thermal variations; at temperatures near the lower end of our recorded range (0–10 °C), the microbial hydrolysis of urea into ammonium (NH_4^+) is significantly retarded. This reduction in the hydrolysis rate subsequently lowers the partial pressure of NH_3 at the soil-air interface, thereby reducing total nitrogen losses during cold spells.

4. FUTURE RECOMMENDATIONS

Based on the findings, the following future research directions are recommended:

It is recommended to investigate the synergy between urease inhibitors and subsurface application to further mitigate nitrogen losses. The efficiency of nano-coated nitrogen fertilizers should be evaluated under the semi-arid climatic conditions of northern Iraq.

Long-term field-scale trials should be conducted to validate these pot-scale results under diverse irrigation management conditions. Predictive models for ammonia volatilization should be developed that integrate real-time soil temperature and moisture sensors for use in precision agriculture.

5. CONCLUSIONS

1. Subsurface placement (3 cm) significantly reduced ammonia loss by 22.83%.

2. Urea was more stable than ammonium salts under the study's temperature conditions.

3. Future research should focus on:

1) Field trials to verify findings under natural conditions;
2) The impact of different nitrogen application rates;
3) Assessing crop nitrogen uptake and yield;
4) The synergistic effect of using nitrification inhibitors combined with deep application.

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