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Stability of stored N-(n-butyl) thiophosphoric triamide (NBPT) treated urea-based fertilizers

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ABSTRACT

The effectiveness of N-(n-butyl) thiophosphoric triamide (NBPT) in reducing ammonia volatilization from urea-based fertilizers has been thoroughly investigated. However, the stability of this inhibitor during storage of NBPT treated urea and urea ammonium nitrate (UAN) needs further investigation. We compared ammonia volatilization from NBPT treated urea (360 mg NBPT kg⁻¹ urea) and UAN (180 mg NBPT L⁻¹ UAN) that were stored at room temperature for 6, 3 and 0 months. We measured ammonia volatilization with cylindrical chambers fitted with acid-charged discs at five times for 21 d. Total ammonia volatilization (measured as a % of applied nitrogen) was significantly greater in untreated urea and UAN (32% to 33%) than those in NBPT treated urea and UAN (6% to 12%). Reduction of ammonia volatilization was not significantly different among NBPT treated urea (73% to 81%) and UAN (63% to 73%) irrespective of storage time. This implies that farmers can mix their urea-based fertilizers with NBPT formulation 6 months prior to fertilization without compromising the ammonia volatilization reducing property of the NBPT.

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Introduction

Ammonia volatilization from surface-applied urea-based fertilizers is a major contributor to atmospheric ammonia contamination (Environment and Climate Change Canada 2017). When urea-based fertilizers are applied to the soil, the urease enzyme hydrolyzes the urea into ammonium and carbonate ions with an increase in soil pH around the urea granule (Bremner 1995). This increase in pH promotes ammonia volatilization from urea-based fertilizers by shifting the equilibrium between ammonium and ammonia toward ammonia. These losses are greatest when urea-based fertilizers are surface-applied to soils with neutral to alkaline pH and low diffusion capacity, under continuous wetting and drying conditions (Chien, Prochnow, and Cantarella 2009; Christianson et al. 1993).

Urease inhibitor containing N-(n-butyl) thiophosphoric triamide (NBPT) has been found to effectively reduce ammonia volatilization from urea-based fertilizers (Sanz-Cobena et al. 2008). The NBPT is commercially available to farmers under different formulation names such as Agrotain® (Koch Agronomic Services LLC, KS), Arborite® (Weyerhaeuser Co., WA) and ARM UTM (Active AgriScience Inc., BC). Studies have shown that coating or mixing urea and urea ammonium nitrate (UAN) with NBPT can reduce ammonia volatilization from urea and UAN by over 50% and it is most effective during the first week of fertilization (Engel, Jones, and Wallander 2011; Frame 2017).

The spring season is a busy period for farmers as a result of pre-planting operations that have to be carried out. Farmers may prefer to buy and mix their urea and urea ammonium nitrate (UAN)

with NBPT formulation at any time during the off growing season and store until application time. The stability of the NBPT with time following its mixing with urea and UAN is important to maximize the benefits of urease inhibitors by farmers. Most studies (Engel, Jones, and Wallander 2011; Frame 2017; Sanz-Cobena et al. 2008) have focussed on the inhibitory effect of urease inhibitors that are mixed with urea-based fertilizers at the time of fertilizer application. Watson et al. (2008) studied the stability of NBPT treated urea and UAN that were stored for 9 months under various storage temperatures (4, 15 and 25 °C). The study reported that the stability of NBPT was correlated to storage temperature as the NBPT recovery was lowest at 25 °C. However, the study of Watson et al. (2008) did not determine the efficacy of stored NBPT treated urea and UAN in reducing ammonia volatilization. A study by Cantarella et al. (2016) compared ammonia volatilization from urea that was freshly treated with NBPT to urea that was treated and stored for various periods of time. The study found that ammonia volatilization in freshly treated urea was not significantly different from NBPT treated urea that was stored for up to 6 months. However, the study of Cantarella et al. (2016) was limited to granular urea fertilizers, and the effect of storage on other urea-based fertilizers such as UAN was not documented. A recent review by Cantarella et al. (2018) suggested that the longevity of NBPT formulations applied to urea depends on the additives including solvent used by different manufactures. ARM U (18% NBPT, m/v) is a relatively new NBPT formulation with a claim by its manufacturer that it contains a polymer (proprietary information) that allows for low application rate of NBPT without compromising its efficacy. Greenhouse and field studies to evaluate the efficacy of ARM U confirmed that a lower NBPT rate with ARM U (360 mg NBPT kg⁻¹ urea) was as effective as a higher NBPT rate with ARM U (540 mg NBPT kg⁻¹ urea), Arborite (480 mg NBPT kg⁻¹ urea) and Agrotain (600 mg NBPT kg⁻¹ urea) (Lasisi, Akinremi, and Kumaragamage 2019). Another claim of ARM U by its manufacturer was its ease of handling at a temperature as low as -15 °C. However, there is a dearth of information on the effect of storage time of NBPT treated urea and UAN on the efficacy of new NBPT formulations such as ARM UTM. Therefore, our study was conducted to determine the effect of storage time of urea and UAN treated with a new formulation of NBPT, ARM U, on the effectiveness of NBPT in reducing ammonia volatilization.

Materials and methods

Soil description and experimental design

This study was conducted in a greenhouse facility at the University of Manitoba, Manitoba, Canada. The soil (0–15 cm) used for this study was a High Bluff series classified as Gley Cumulic Regosol (Manitoba Soil Survey 1972). The soil texture (Gee and Bauder 1986) is a loam (sand is 450 g kg⁻¹, silt is 310 g kg⁻¹ and clay is 240 g kg⁻¹) with organic matter (Walkley and Black 1934) of 47 g kg⁻¹, inorganic nitrogen (N) of 7.4 mg kg⁻¹ (Maynard, Kalra, and Crumbaugh 2008), volumetric water content at field capacity (Cassel and Nielsen 1986) of 0.37 m³ m⁻³ and soil pH_{water} (1:1) of 7.7.

The experimental setup was a randomized complete block design with four replications. Two sources of N, urea (46-0-0) and UAN (28-0-0), were used for this study. There were nine fertilizer treatments which were urea treated with NBPT at 6 (UR₆), 3 (UR₃) and 0 (UR₀) months before the start of the study; UAN treated with NBPT at 6 (UAN₆), 3 (UAN₃) and 0 (UAN₀) months before the start of the study; untreated urea, untreated UAN and a control (bare soil) with neither urea nor UAN. The NBPT formulation used was ARM UTM containing 18% NBPT m/v as an active ingredient. Urea was coated with ARM U at 2 L per 1000 kg urea and ARM U was mixed with UAN at 1 L per 1000 L UAN. The coating of ARM U with urea granule gave a concentration of 360 mg NBPT kg⁻¹ urea while the mixing of ARM U with UAN gave a concentration of 180 mg NBPT L⁻¹ UAN which is an equivalent of 360 mg NBPT kg⁻¹ urea (based on urea fraction of UAN). The NBPT treated urea and UAN were stored at room temperature until the start of the experiment. The UR₀ and UAN₀ were prepared a day before the start of the study.



Chamber installation and ammonia measurement

Ammonia volatilization was measured with cylindrical chambers (20 cm in height by 15 cm i.d) following the method described by Grant et al. (1996) and Lasisi, Akinremi, and Kumaragamage (2019). Briefly, the bottoms of the chambers were sealed with flat plastic plates followed by filling the chambers with soil up to 5 cm at a bulk density of 1.1 Mg m^{-3} . Water was added to the soil in the chambers to bring the water content to 75% field capacity. The chambers were covered with plates for 24 h for the water to equilibrate after which treatments (100 kg N ha^{-1}) were applied to the center of the chambers. This was immediately followed by tightly fitting the chambers with acid-charged discs at 5 cm above the soil surface (lower disc) and 2 cm from the top of the chamber (upper disc). The discs were polyethylene foam with a mass of 24.5 g, thickness of 2.5 cm and diameter of 16 cm. The discs were designed to tightly fit the chambers. The discs were charged by thoroughly rinsing and wringing in 0.001 M sulfuric acid and then glycerol-phosphoric acid solution (40 mL 14.7 M phosphoric acid and 50 mL glycerol in 1 L distilled water). The lower discs trapped volatilized ammonia from the soil surface and the upper disc prevented atmospheric contamination of the lower disc.

The acid-charged discs were sampled and replaced on 2, 4, 7, 14 and 21 dafter fertilizationto measure the volatilized ammonia. Each lower disc was placed in a pre-labeled zip-lock bag and transferred to the laboratory for extraction on the same day. After sampling, the chambers were weighed to determine moisture loss on 4, 7 and 14 d and the weight difference was made up by adding the required amount of water. Daily mean air and soil temperatures were monitored with Watchdog 2000 series weather station (model 146 2900ET; Spectrum Technologies Inc., IL).

In the laboratory, the amount of acid solution in the lower disc was determined by weighing the disc before it was extracted for ammonia. Ammonia trapped in the disc was extracted by dispensing 250 mL of 0.5 M potassium chloride (KCl) on the disc in the zip-lock bag. This disc in the zip-lock bag was squeezed thoroughly and the aliquot was decanted into a vial. The aliquot of the resulting solution was stored in the refrigerator (4°C) and analyzed within 2 d of extraction for ammonium-N concentration using AQ2 Discrete Analyzer (SEAL Analytical Inc., WI, USA).

Ammonia volatilization was calculated as follows:

$$NH_3 \text{ loss } (kg \text{ N ha}^{-1}) = \frac{(\text{Extractant (mL)} + \text{absorbent in disc (mL)}) \times NH_3 (mg \text{ N mL}^{-1})}{\text{Area of chamber (ha)} \times 10^6}$$

Cumulative ammonia volatilization was calculated by summing ammonia volatilization measured between sampling days. Total ammonia volatilization (measured as a % of applied N) was calculated by summing ammonia volatilization measured between sampling days for the 21 d after deducting ammonia volatilization from bare soil.

At the end of the study (21 d), soil in each chamber was thoroughly mixed. Six grams of each soil was sampled and extracted with 25 mL of 2 M KCl to determine ammonium and nitrate concentrations (Maynard, Kalra, and Crumbaugh 2008). Also, soil samples were collected from each chamber to determine the gravimetric moisture content.

Statistical analysis

GLIMMIX procedure for repeated measure analysis in SAS 9.4 (SAS Institute Inc. 2014) was used to determine the effect of treatments and day on cumulative ammonia volatilization. In the GLIMMIX model, treatments and time were fixed effects and block (replicate) is a random effect. GLIMMIX procedure was also used to determine the effect of treatments on total ammonia volatilization and ammonium and nitrate concentrations remaining in the soil (after deducting inorganic N from the bare soil) at the end of the study. Prior to using the GLIMMIX procedure, data were checked for the assumption of normal distribution (Shapiro-wilk test). All data except nitrate concentrations were lognormally distributed and a lognormal distribution was specified in their models. The effect of

storage time of NBPT treated urea and UAN on percentage reduction of ammonia volatilization was tested with GLIMMIX procedure (beta distribution). Treatment mean comparison was performed with Fisher's Protected least significance difference (LSD) at a probability level (P) of 0.05.

Results and discussion

Daily mean air temperature ranged from 13.4 °C to 24.7 °C (Figure 1). Similarly, daily mean soil temperature ranged from 14.4 °C to 24.5 °C (Figure 1). Except for 12 d, the daily mean air and soil temperatures were above 17 °C.

Cumulative ammonia volatilization

There was a significant treatment by time interaction on cumulative ammonia volatilization ($P = 0.0017$). Cumulative ammonia volatilization was significantly greater in untreated urea and UAN than NBPT treated urea and UAN throughout the sampling period (Figure 2). Cumulative ammonia volatilization was not significantly different between the untreated urea and UAN throughout the study. When the ammonia volatilization was compared based on the source of N, cumulative ammonia volatilization among NBPT treated urea was significantly different from 2 to 7 d but not after 7 d (Figure 2). Cumulative ammonia volatilization from UR_6 was significantly greater than cumulative ammonia volatilization in UR_0 from 2 to 7 d but not greater than cumulative ammonia volatilization from UR_3 . After 7 d, cumulative ammonia volatilization among the NBPT treated urea was not significantly different. In the case of UAN source, cumulative ammonia volatilization among the NBPT treated UAN was not significantly different throughout the sampling period.

Greatest ammonia volatilization from untreated urea ($16.9 \text{ kg N ha}^{-1}$) was measured on 4 d which resulted in cumulative ammonia volatilization of 20.4% of applied N from 0 to 4 d (Figure 2). In contrast, the greatest amount of ammonia volatilization in NBPT treated urea (4.5 to 6.7 kg N ha^{-1}) was measured on 14 d to give cumulative ammonia volatilization of 5.5% to 8.5% of applied N from 0 to 14 d (Figure 2). With the UAN treatments, the greatest ammonia volatilization from untreated UAN ($10.1 \text{ kg N ha}^{-1}$) was measured on 14 d to give cumulative ammonia volatilization of 31.3% of applied N from the day the treatment was applied. In the case of NBPT treated UAN, the greatest amount of ammonia volatilization also measured on 14 d (2.8 to 3.6 kg N ha^{-1}) to give cumulative ammonia volatilization of 8.0% to 10.5% of applied N from 0 to 14 d (Figure 2). The time of greatest ammonia volatilization among the treatments

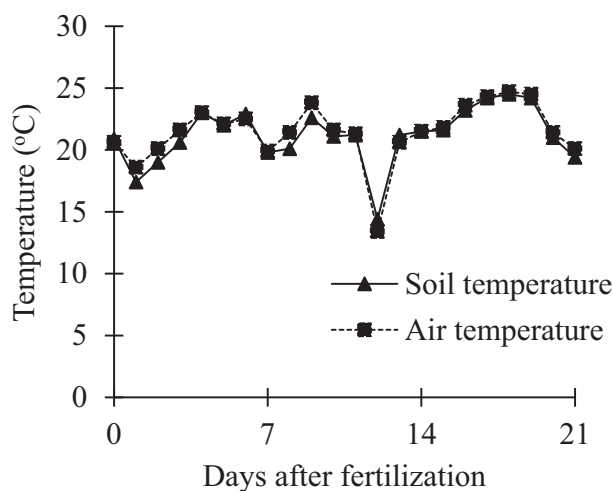


Figure 1. Daily mean air and soil (at 2 cm) temperatures during the study.

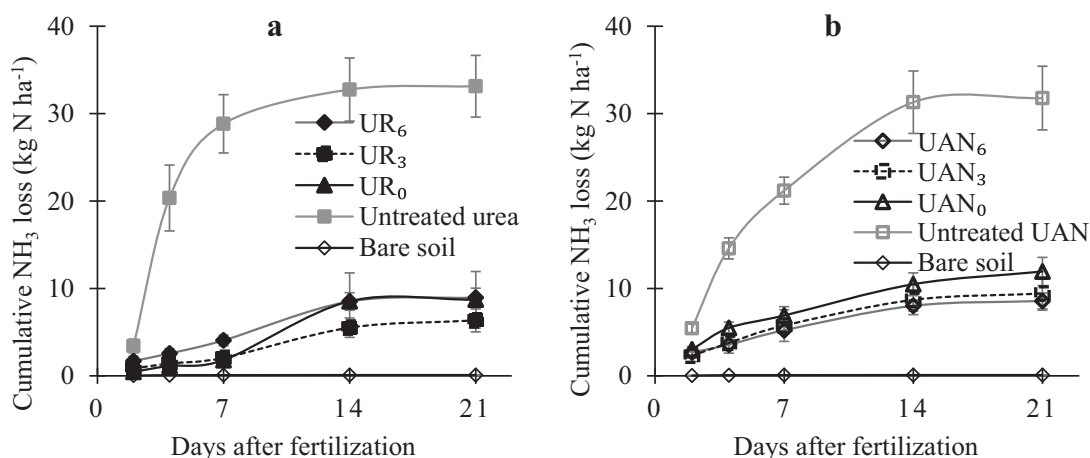


Figure 2. Cumulative ammonia volatilization following surface application of (a) urea and (b) UAN treated with and without NBPT. Error bars are standard errors of the mean.

Note: UR₆, UR₃ and UR₀ are stored NBPT treated urea for 6, 3 and 0 months, respectively; UAN₆, UAN₃ and UAN₀ are stored NBPT treated UAN for 6, 3 and 0 months, respectively; NBPT is N-(n-butyl) thiophosphoric triamide.

was similar to other studies (Engel, Jones, and Wallander 2011; Viero et al. 2014) that showed ammonia volatilization peaked within the first week of fertilization in untreated urea and after the first week of fertilization in NBPT treated urea and UAN.

Total ammonia volatilization

There was a significant effect ($P < 0.0001$) of treatment on total ammonia volatilization (Table 1). Untreated urea and UAN lost significantly greater amounts of ammonia than NBPT treated urea and UAN at the end of the study (Table 1). Total ammonia volatilization was not significantly different among NBPT treated urea and UAN irrespective of the time of coating or mixing with NBPT (Table 1). Total volatilized ammonia in NBPT treated urea ranged from 6.3% to 8.9%, while it ranged from 8.5% to 11.8% in NBPT treated UAN. Conversely, total ammonia volatilization was 33.0% in untreated urea and 31.7% in untreated UAN. More than 55% of the total ammonia volatilization that was measured in NBPT treated urea occurred during the second and third week period (Figure 2). Unlike the NBPT treated urea, 87% of total ammonia volatilization in untreated urea occurred within 1 week after fertilization. This agrees with other studies that showed that most of the ammonia volatilization in untreated urea occurred during the first week of fertilization whereas it occurred after the first week of fertilization in NBPT treated urea (Goos

Table 1. Total ammonia volatilization, percentage reduction of ammonia volatilization, soil ammonium (NH₄⁺) and nitrate (NO₃⁻) concentrations from surface-applied urea and UAN treated with and without NBPT.

Treatment	Total ammonia (%)	Reduction (%)	Soil NH ₄ ⁺ (mg N kg ⁻¹)	Soil NO ₃ ⁻ (mg N kg ⁻¹)
Untreated urea	33.0 a		2.5 a	117.1 b
UR ₆	8.9 bc	73 ab	4.5 a	145.2 ab
UR ₃	6.3 c	81 a	8.3 a	117.1 b
UR ₀	8.6 bc	74 ab	2.6 a	168.5 a
Untreated UAN	31.7 a		3.5 a	154.9 ab
UAN ₆	8.5 bc	73 ab	9.5 a	178.9 a
UAN ₃	9.3 bc	71 ab	12.0 a	167.4 a
UAN ₀	11.8 b	63 b	8.3 a	171.3 a

Note: UR₆, UR₃ and UR₀ are stored NBPT treated urea for 6, 3 and 0 months, respectively; UAN₆, UAN₃ and UAN₀ are stored NBPT treated UAN for 6, 3 and 0 months, respectively; NBPT is N-(n-butyl) thiophosphoric triamide. Means with different letters within a column are significantly different at $P < 0.05$ LSD mean separation.

2013; Tian et al. 2015). In addition, the quantity of total ammonia volatilization from NBPT treated and untreated urea in our study was similar to the total ammonia volatilization measured by Frame (2017).

Similar to total ammonia volatilization, there was no significant effect of storage time among NBPT treated urea as well as among NBPT treated UAN on the effectiveness of NBPT in reducing ammonia volatilization (Table 1). Treating urea and UAN with NBPT reduced ammonia volatilization by 73% to 81% in urea and by 63% to 73% in UAN at the end of 21 d, regardless of when the treatment took place. The amount of ammonia volatilization reduction from urea and UAN due to NBPT from this study was similar to previous studies that reported over 50% less ammonia volatilization from urea and UAN with NBPT (Engel, Jones, and Wallander 2011; Turner et al. 2010). The lack of significant differences in the percentage reduction of ammonia volatilization from stored NBPT treated urea and UAN showed that NBPT retained its effectiveness in reducing ammonia volatilization irrespective of the time of storage for up to 6 months after treatment. Our result from stored NBPT treated urea in this study was similar to that of Cantarella et al. (2016) that found no significant difference in ammonia volatilization between fresh NBPT treated urea and stored NBPT treated urea up to 6 months. Although Watson et al. (2008) reported the half-life of NBPT in NBPT treated urea to be 74 and 188 d at 25 °C and 15 °C, respectively, this reported degradation did not have any significant effect on the effectiveness of stored NBPT in reducing ammonia volatilization in our study.

Soil ammonium and nitrate concentrations

There was no significant effect ($P = 0.7471$) of treatments on ammonium concentrations among treatments (Table 1). Despite UAN_3 having the highest ammonium concentration ($12.0 \text{ mg N kg}^{-1}$), its ammonium concentration was not significantly different from those in untreated urea and UR_0 (2.6 mg N kg^{-1}). In contrast, there was a significant treatment effect ($P = .0243$) on the concentration of nitrate. Among the urea treatments, UR_0 had the highest nitrate concentration ($168.5 \text{ mg N kg}^{-1}$) while untreated urea and UR_3 had the smallest nitrate concentrations ($117.1 \text{ mg N kg}^{-1}$). Nitrate concentrations were not significantly different among the UAN treatments.

The expectation was for soil available N concentration to increase as percentage reduction of ammonia volatilization increases or as total ammonia volatilization decreases. However, the soil available N of some treatments did not reflect these expected concentrations on a statistical basis (Table 1). The lack of significant difference in available N is possibly due to variability in the ammonium concentrations (coefficient of variation ranged from 73% to 144%, $\text{LSD} = 11.1$). The inconsistency between the total ammonia volatilization and soil available N concentrations is not unique to this study. Previous studies that measured ammonia volatilization and soil available N concentrations from urea treated with and without NBPT and nitrification inhibitor found recovered soil available N concentrations to be higher in some plots with higher ammonia volatilization than where ammonia volatilization was lower (Frame 2017; Soares, Cantarella, and Menegale 2012). It is possible that the measurement of other pathways of N disappearance from the available pool such as denitrification and immobilization may account for this inconsistency. Potential N losses due to immobilization (if any) will later be mineralized to become available N. In contrast, N losses in form of nitrous oxide emission during nitrification and denitrification neither present any agronomic benefit to the farmers nor represent a significant portion of applied N. Nitrous oxide emission is an average of 1% of applied N according to the Intergovernmental Panel on Climate Change (2006). However, nitrous oxide emission has a serious negative environmental consequence (Harty et al. 2016).

Conclusion

The new NBPT formulation, ARM UTM, significantly reduced ammonia volatilization from urea and UAN. Reduction of ammonia volatilization among NBPT treated urea ($360 \text{ mg NBPT kg}^{-1} \text{ urea}$) and UAN ($180 \text{ mg NBPT L}^{-1} \text{ UAN}$) was not significantly different for treatments that were stored at room

temperature for periods that ranged from 0 to 6 months. This study shows that farmers can treat their urea-based fertilizers with NBPT formulation at least 6 months prior to application and store at room temperature without compromising the effectiveness of NBPT. On the other hand, producers of NBPT formulations can coat urea fertilizers and sell these to farmers without compromising its efficacy.

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