

Reduction of Ammonia Loss by Mixing Urea with Liquid Humic and Fulvic Acids Isolated from Tropical Peat Soil

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Abstract: Problem statement: Fertilizer N use efficiency is reduced by ammonia volatilization. Under low soil CEC and high pH, N from soil solution is released to the atmosphere. Ammonia loss due to low worldwide N use efficiency (33%) has been implicated in global warming. Thus, the objectives of this laboratory study were to evaluate the effectiveness of liquid humic and fulvic acids, isolated from tropical peat soils in reducing N loss from urea fertilizer as well as to investigate the ability of these acids to retain NH_4^+ and NO_3^- or reduce soil pH. **Approach:** Formulated liquid N fertilizers consisting of urea and different types of humic molecules (HA or FA or mixture of both), solid and liquid urea were surface applied to 250 g of soil. A closed dynamic air flow system was used to trap NH_3 loss in boric acid after which samples were titrated with 0.01 M HCl to estimate NH_3 loss. After 30 days of incubation, the soil was air dried and analysed for pH, exchangeable NH_4^+ , available NO_3^- and exchangeable cations. The results were analysed using SAS and treatments means were compared using Duncan's New Multiple Range Test (DNMRT). **Results:** The use of humic molecules reduced NH_3 loss and increased exchangeable NH_4^+ . The high CEC of Humic Acids (HA) made the LHA treatment the best in reducing N loss after surface application. The presence of HA and Fulvic Acids (FA) increased NH_4^+ recovery. Even though, the soil pH of all the treatments were high, significant reduction of N loss was observed for humic molecules treatments. **Conclusion:** The use of liquid organic N fertilizer has the ability to reduce NH_3 volatilization in acid soil. The use of both humic and fulvic acids could be effective in promoting NH_4^+ retention. Thus, it can be concluding that, humic substances, in general, have great ability in controlling NH_3 loss and retaining NH_4^+ in acid soils. It could be a cheapest, practical and easiest way to control N loss.

Key words: Ammonia volatilization, humic acids, fulvic acids, liquid N fertilizers, ammonium, nitrate

INTRODUCTION

Ammonia (NH_3) loss from volatilization processes reduces urea-N fertilizer use efficiency^[1,2]. According to Raun and Johnson^[3], worldwide N Use Efficiency (NUE) was approximately 33%. This low value suggests the environmental problems associated with the use of unbalanced N fertilizers. In considering the economic aspect, 67% loss was estimated to cost USD 15.9 billion. This huge amount of money does not include that needed in maintaining a clean environment with the existence of problems created due to unbalanced use of N fertilizers.

Under normal conditions, ammonium (NH_4^+), hydroxyl (OH^-) and carbonate (CO_3^{2-}) ions are produced rapidly (1 or 2 days) after surface application of urea by urease^[4]. This leads to the accumulation of NH_4^+ , which simultaneously increases the soil pH surrounding the application area or near the urea granule^[1]. The increase of OH^- , HCO_3^- and NH_4^+ concentrations through this process plays a significant role in the rapid loss of N. Rapid liberation of ammoniacal N makes it more fragile, which promotes soluble N (e.g., NH_4^+) to react with other components in the soil solution and releases NH_3 gas to the atmosphere^[5]. Global estimation of N loss from urea

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alone has been estimated at 9 Tg N year⁻¹^[6]. While, overall losses from all possible sources reaches up to 54 Mt (or 10⁻¹² g) NH₃-N year⁻¹^[7]. It must be stressed that, huge losses of N could be one of the critical environmental issues that contribute to global warming.

There are many factors involved in NH₃ volatilization, which can be grouped into soil (e.g., pH, CEC), environment (e.g., temperature, humidity) and management (e.g., surface application, drilling). A number of studies with different approaches has reduced NH₃ loss^[1,2,8-11]. Acidic materials alone, organic and inorganic additives, mixture of acidic materials and additives could reduce N loss by 60, 38.5 and 49%, respectively^[1,2,9-11]. Generally, the main concerns for the above mixtures are that they create an acidic environment from acidic materials used and inhibit ureolytic microorganisms' activities which in effect slows down the release of NH₄⁺ into the soil and indirectly reduces N loss^[12].

In this regard, a new or better fertilizer formulation needs to be developed from organic based materials, which have the capability to retain NH₄⁺ (due to high CEC) as well as maintaining low soil pH. Thus, the objectives of this laboratory study were to evaluate the effectiveness of liquid humic and fulvic acids, isolated from tropical peat soils, in reducing N loss from urea fertilizer as well as to investigate the ability of these acids to retain NH₄⁺ and NO₃⁻ or reduce soil pH.

MATERIALS AND METHODS

Mineral and peat soil samples were collected at 0-25 cm depth from minimal disturbed area of Universiti Putra Malaysia Bintulu Campus, Sarawak and Kuala Tatau, Sarawak, Malaysia respectively. The mineral soil was a sandy clay loam Typic Paleudults (Nyalau series) and the peat soil was Sapristis (well decomposed humic matter). Both soils were air dried, ground and sieved to pass through 2 mm size.

The soil pH was analyzed using a glass electrode, organic carbon by combustion method^[12], CEC by leaching with ammonium acetate at pH 7 before steam distillation^[13] and exchangeable cations (K, Ca and Mg) using Atomic Absorption Spectrophotometry (AAS). Prior to treatments preparation, humic acid (HA) was isolated according to the method of Susilawati *et al.*^[14]. Extraction time of 4.5 h using a mechanical shaker at 188 rpm, acidification with 6 N HCl, fractionation under room temperature for 2 h and centrifugation time of 15 min were adopted for preparation of liquid humic acid (LHA) and Liquid Fulvic Acid (LFA). The mixture of LHA+LFA (acidified) and LHA+LFA (unacidified)

were prepared by including or excluding the acidification step and without centrifugation. All treatments had a ratio of 4:50 [urea (g): Distilled water (mL)]. Treatments evaluated in this study were; T0-soil alone, T1-urea (liquid), T2-urea (solid), T3-LHA+Urea, T4-LFA+urea, T5-LHA+LFA (acidified)+urea, T6-LHA+LFA (unacidified)+urea and (T7) ammonium sulphate (liquid).

Ammonia volatilization from the formulated fertilizers was evaluated in a laboratory incubation experiment. A closed dynamic air flow system with modification was used to measure the volatilized NH₃^[15]. The system consisted of: (1) Air pump with the flow rates ranging from 1-2 L min⁻¹ and (2) An exchange chamber (500 mL Erlenmeyer flask) and a trap (250 mL Erlenmeyer flask), both stoppered and fitted with an inlet/outlet. An inlet was connected to the chamber with air pump, whilst the outlet was connected to the trap containing boric acid using polyethylene tube. A 250 g of mineral soil was placed in the exchange chamber to a depth of 3 cm. The soils were then moistened with liquid organic fertilizers (as treatments) or liquid urea and/or distilled water up to 75% field capacity. The moisture was maintained throughout the experiment by weighing the samples daily and distilled water was used to top up the difference.

Ammonia was collected in boric acid by air flow circulation, which passed through the exchange chamber into the trapping flask containing 100 mL boric acid with bromocresol green and methyl red indicator. It was then titrated with 0.01 M HCl to estimate the NH₃ released. The data was collected every 24 h for 30 days. At the end of the experiment, the soil samples were removed and air dried. The soil samples were then analyzed for pH (water and KCl), exchangeable NH₄⁺ and NO₃⁻ and cations (double acid method) (K, Ca and Mg) using atomic absorption spectrometer (AAS-800). The experiment was conducted using a Completely Randomized Design (CRD), with three replications. Treatment effects were assessed using analysis of variance while treatment means were compared using Duncan's New Multiple Range Test (DNMRT). Statistical Analysis System (SAS) version 9.1 was used for the statistical analysis.

RESULTS

The mineral soil (Nyalau series) pH was acidic in water and KCl (Table 1). The soil contained 0.26% N, 2.99% total organic carbon and 5.16% organic matter. Low exchangeable cations (K, Ca and Mg) was recorded (Table 1). The CEC was 24.5 cmol kg⁻¹.

These information are consistent with those reported by Paramananthan^[16].

Table 1: Selected chemical properties of nyalau series

Property	Values
pH _w	4.040
pH _{KCl}	3.490
Exchangeable K ⁺ (cmol kg ⁻¹)	0.113
Exchangeable Ca ²⁺ (cmol kg ⁻¹)	1.002
Exchangeable Mg ²⁺ (cmol kg ⁻¹)	1.001
CEC (cmol kg ⁻¹)	24.500
Total nitrogen (%)	0.260
Total organic carbon (%)	2.990
Organic matter (%)	5.160
C/N ratio	11.500

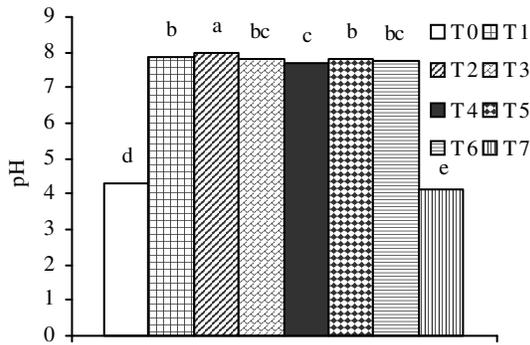


Fig. 1: Soil pH_w after 30 days of incubation. (Different letter indicate significant difference between means using Duncan's New Multiple Range Test (DNMRT) at p = 0.05)

The pH (water and KCl) recorded for treated soils were significantly different between treatments (Fig. 1 and 2). Soil treated with T1 or T2 gave higher pH_w value as compared to other treatments; the highest was noted for T2. T5 was the third highest. Statistically it was not different from T1. As compared to T0 and T7, the pH_w of the treated soil with urea (T1 and T2) and organically based liquid N fertilizers (T3 to T6) was significantly higher. Interestingly, T3 gave almost similar pH_w values with T6; and these two treatments only showed their differences from T2, T0 and T7. Even though T3 gave the highest pH value (9.04) during liquid organic fertilizer formulation (Table 2), its effect was temporary. After 30 days of incubation, the pH recorded for T3 was among the lowest, among organically based liquid N fertilizers treatments. The lowest pH_w was recorded for T7 treatment followed by T0 and T4. In considering the effect of humic substances in reducing pH_w, treated with urea, LFA was better among liquid organic N fertilizer treatments.

In KCl solution, the treatments gave different results. The highest pH_{KCl} was recorded for T5, followed by T1, T2 and T4 (Fig. 2). These four

treatments were not significantly different. However, they were significantly different from T6 and T3.

Table 2: Average pH values of formulated liquid fertilizers

Code	Treatment	pH	
		Without urea	With urea
F1	Urea (liquid)	Nd	8.14
F2	HA	1.89	9.04
F3	FA	1.02	1.64
F4	HA+FA (acidified)	0.98	1.60
F5	HA+FA (unacidified)	6.56	6.89

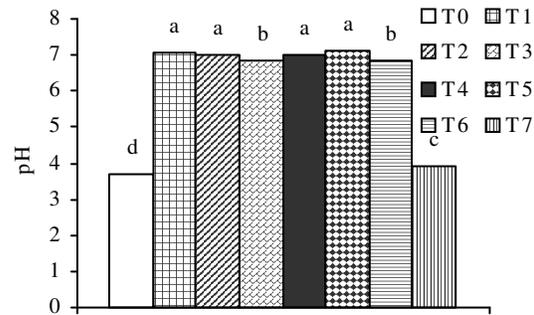


Fig. 2: Soil pH_{KCl} after 30 days of incubation. (Different letter indicate significant difference between means using Duncan's New Multiple Range Test (DNMRT) at p = 0.05)

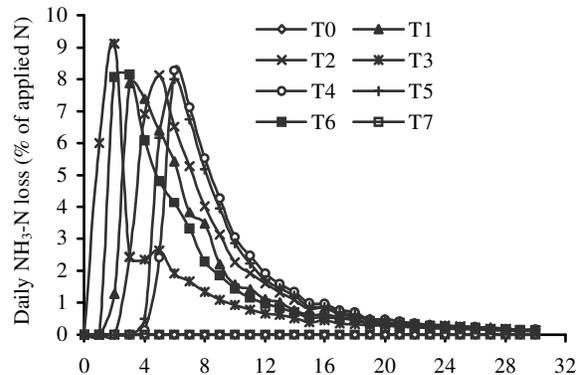


Fig. 3: Daily loss of ammonia for 30 days of incubation

As compared to T0 and T7, all the treatments with liquid organic based N fertilizers showed higher and significant results.

Daily NH₃ loss from T1 and T2 was maximum at day 3 (7.89%) and day 5 (8.12%), respectively (Fig. 3). T4 and T5 recorded similar peaks of NH₃ loss (6 days after treatments application). However, T6 and T3 showed a different trend.

T4 delayed N loss by 1-2 days as compared to T2 and T1, respectively. Similar result was noted for T5 treatment. T6 had no effect in delaying the N loss. In

the case of T3 treatment, N loss occurred a day earlier compared to T2 and 2 days earlier compared to T1.

Table 3: Cumulative NH₃ loss for 30 days of incubation

Label	Treatment	N loss (%)	Reduction obtained as compared to T2 (%)
T0	Control	0 ^c	nd
T1	Urea (liquid)	48.74 ^{ab}	3.88
T2	Urea (solid)	50.71 ^a	nd
T3	LHA plus urea	35.90 ^c	29.21
T4	LFA plus urea	44.91 ^b	11.44
T5	LHA+LFA (acidified)+urea	46.60 ^{ab}	8.11
T6	LHA+LFA (unacidified)+urea	48.65 ^{ab}	4.06
T7	Ammonium sulphate (liquid)	0 ^d	100

Different letter indicate significant difference between means using Duncan's New Multiple Range Test (DNMRT) at p = 0.05; *nd = Not Determine

Table 4: Soil exchangeable NH₄⁺ and available NO₃⁻ contents for 30 days of incubation

Label	Treatment	NH ₄ ⁺ (mg kg ⁻¹)	NO ₃ ⁻ (mg kg ⁻¹)
T0	Control	12.60 ^d	10.27 ^c
T1	Urea (liquid)	170.80 ^c	12.13 ^{bc}
T2	Urea (solid)	170.80 ^c	12.13 ^{bc}
T3	LHA plus urea	179.67 ^c	11.20 ^{bc}
T4	LFA plus urea	224.00 ^b	12.13 ^{bc}
T5	LHA+LFA (acidified)+urea	217.00 ^b	18.67 ^{ab}
T6	LHA+LFA (unacidified)+urea	172.20 ^c	12.13 ^{bc}
T7	Ammonium sulphate (liquid)	774.20 ^a	21.00 ^a

Different letter indicate significant difference between means using Duncan's New Multiple Range Test (DNMRT) at p = 0.05

The minimum period for maintaining significant N loss (1% from added N, as urea) was different for all the treatments. Generally effects of organically based N fertilizers were different, vis a vis organic molecules type used in fertilizer formulation. In this study, presence of LHA (T3) required a minimum of 10 days to reach 1% N loss, whilst T6 required 12 days. Thirteen and 15 days were recorded for T1 and T2, respectively.

In general, the used of organic additives (e.g., LHA or LFA separately or their mixture) significantly reduced N loss (Table 3). Significant reduction of cumulative N loss, of 29.21% was recorded for T3 followed by T4 (Table 3). Other liquid organic N fertilizers treatments gave no significant effect as compared to T1.

Exchangeable NH₄⁺ and NO₃⁻ content in the soil were significantly different among treatments. The highest NH₄⁺ recovery was recorded for T7 treatment (Table 4). It was followed by T4 and T5. Lowest NH₄⁺ recovery was recorded for T1 and T2 followed by T3. Other organic additives did not result in any significant difference as compared to T1 and T2. The effect of T0, T1, T2, T3, T4, T5 and T6 on soil available NO₃⁻ was

not significantly different from T7 (Table 4). The NO₃⁻ was generally low in the soil after 30 days of incubation (Table 4).

Table 5: Soil exchangeable K, Ca, Mg and Na for 30 days of incubation

Label	Exchangeable cations (cmol kg ⁻¹)			
	K	Ca	Mg	Na
T0	0.119 ^c	0.754 ^a	0.715 ^a	0.138 ^b
T1	0.113 ^c	0.565 ^b	0.399 ^{cd}	0.120 ^b
T2	0.117 ^c	0.592 ^b	0.376 ^d	0.121 ^b
T3	0.127 ^c	0.588 ^b	0.412 ^{bcd}	0.137 ^b
T4	0.199 ^b	0.628 ^b	0.419 ^{bc}	0.126 ^b
T5	0.221 ^a	0.633 ^b	0.445 ^b	0.130 ^b
T6	0.215 ^{ab}	0.640 ^b	0.407 ^{bcd}	0.135 ^b
T7	0.123 ^c	0.817 ^a	0.684 ^a	0.333 ^a

Different letter indicate significant difference between means using Duncan's New Multiple Range Test (DNMRT) at p = 0.05

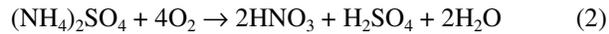
Increase of exchangeable K was recorded for all of the liquid organic N fertilizers (Table 5). As compared to T1, T4, T5 and T6 gave a range of 76 to 95% increase in exchangeable K. Other treatments [including (NH₄)₂SO₄] gave no significant results, as compared to T0 or T1 and T2. Exchangeable Ca and Mg from T1, T2, T3, T4, T5 and T6 treatments were lower compared to T0 and T7. They did not show any statistical difference among them. The highest content of exchangeable Na was noted for T7 treatment. It was significantly different from those of T0, urea (T1 and T2) and all organically based liquid N fertilizers (T3 to T6). The Na contents of the other treatments were only significantly different from that of T0.

DISCUSSION

As expected, solid form of urea resulted in higher pH_w value compared to other treatments. This was probably due to hydrolysis processes that occurred just after application of urea fertilizer. Hydrogen released mainly by hydrolysis of Al, non-exchangeable hydroxyl Al and Fe and dissolution of organic matter, could be used during urea hydrolysis. This process generates OH⁻ which accumulate not only at the microsite but completely in the soil [17]. This may have contributed to the increase of pH. Even though some of the fertilizer mixtures [e.g; T5 (1.60), T4 (1.64)] (Table 2), used in this study were acidic, the pH increased sharply after 30 days of incubation. Higher quantity of H⁺ (due to HCl during fractionation stage) in these mixtures could have facilitated urea hydrolysis and promoted the production of OH⁻.

Ammonium sulphate was the effective fertilizer in reducing the soil pH because N fertilizer, in the form of NH₄⁺ has the ability to react during nitrification in soils

(equations 1 and 2) to produce acidic residues in the form of H^+ , NO_3^- and even SO_4^{2-} . According to Foth and Ellis^[18], almost 12 kg of $CaCO_3$ will be needed in recovering the acidity by $(NH_4)_2SO_4$ in 1 kg soil:



The CEC provided by HA, which ranged between 417-583 $cmol\ kg^{-1}$ (based on our previous study) may have contributed to ammonia loss reduction. The negative sites due to ionization of carboxylic (COOH) and phenolic (OH) might have improved NH_4^+ retention hence reduction in N loss^[19]. These negative charges could develop with the level of salt and pH, that occurred in soil^[20,21]. More salt will produce more negative charge in soil. A similar situation will occur at high pH. Thus, the presence of KOH (residues by extraction procedure), as a source of salt, could enhance HA charges and indirectly reducing the N loss.

The high pH recorded for water and KCl could be another factor for the efficient use of humic molecules in controlling N loss, due to high pH recorded and could increase the CEC^[21,22]. Humic molecules have the ability to retain cations at their surfaces. The retention of NH_4^+ might be a factor for the low available NO_3^- in soil. Reduction conversion of NH_4^+ to NO_3^- would then occur due to lack of NH_4^+ in soil solution. A similar observation has been reported by Ahmed *et al.*^[23].

The information on exchangeable Ca and Mg supports NH_3 loss reduction. The increase in these two cations suggests that there was some replacement and retention of NH_4^+ in the soil treated with liquid organic fertilizers^[23]. Even though, no statistical difference was observed, the trend of NH_3 loss provides early information on the relationship between NH_4^+ retention, reduction of N loss and exchangeable cations.

T7 caused the highest exchangeable Ca, Mg and Na in this study. This was expected for NH_4^+ -N fertilized soils. Since the exchange sites of soils saturated with cations such as Ca, Mg, Na and K in the order of $Ca > Mg > K > Na$, more NH_4^+ in particular is expected to be in soil solution for T7^[18].

Higher exchangeable K recorded in some of the treatments was probably due to higher content of K associated with them. Based on our analysis, the content of K in fulvic acid solution was high, almost 2000 ppm in 50 mL. This value was 20 times higher as compared to LHA [previous paper, almost 100 ppm in 50 mL (after 1st washing)]. In the case of T4,

splitting LHA and LFA reduced the K content in LFA solution and this could be the reason why LFA gave lower exchangeable K as compared to T5 and T6 treatments.

CONCLUSION

Liquid organic N fertilizer has the ability to reduce NH_3 volatilization in an acid soil. Even though, the amount used was small as compared to the quantity of soil used, the effect on NH_3 loss cannot be ignored. HA was the most effective material that effectively controlled NH_3 loss. The use of both humic and fulvic acids is effective in promoting NH_4^+ retention. Thus, it can be concluded that, humic substances, in general, have a great ability in controlling NH_3 loss and retaining NH_4^+ in an acid soil. It could be one of the cheapest, easiest and practical ways in controlling N loss. However, the rate or amount that is most effective together with its possibility to be used as foliar fertilizer needs to be investigated in detail in future research.

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