Running title: Liquid urea application for better N management

**Nitrogen Dynamics In Tropical Soils Applied With Liquid And Granular Urea Fertilizers**

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 **Novelty statement:**

Liquid urea (LU) has been hardly ever evaluated for N mineralization in comparison to granular urea (GU). Results show that liquid source of N decrease N loss and increase nitrogen use efficiency (NUE). We observed that LU had faster N mineralization than GU while at same application rates which will able to quicker N supply to the plant.

**Abstract**

The mineralization of urea fertilizer mostly regulates the nitrogen dynamics in the soil. This study was conducted in a laboratory to compare the nitrogen dynamics in two tropical soil series incubated with either liquid urea (LU) or granular urea (GU) at 0, 300, 400 or 500 mg/kg of soil for four weeks. The soil series were Bungor and Selangor having sandy clay loam and clay texture, respectively. The NH4+-N, NO3--N concentration in the soils were measured throughout the incubation period. At the same application rate, higher NH4+-N and NO3**-**-N concentrations were recorded in the LU applied soils throughout the incubation period. Urea-N recovery was higher in GU than LU treated soils in the first two weeks while no urea-N was present in both GU and LU treated soils after the third week of incubation. The results suggest that the LU was a better N fertilizer source than GU for a rapid mineralization and quicker availability of N.

**Keywords**: ammonium-N; nitrate-N; liquid urea; total N; NH3 volatilization.

**Introduction**

Nitrogen (N) is an essential element for plants and it is a major constituent of chlorophyll, amino acids, adenosine triphosphate (ATP) and genetic materials and contributes 30-50% of crop yield increases worldwide. Urea is the main N fertilizer source having significant impact to the global crop production (Kira et al., 2019). It is the most widely used N fertilizer because it is user-friendly and having high N content (46%). Urea use covers around 73.4% of total N fertilizer utilization globally (Sutton et al., 2011; Heffer and Prud’homme, 2016). However, the alarming problem associated with the granular urea fertilizer use is its high nitrogen loss and low nitrogen use efficiency (NUE) which ranges from 10-50% (Almaz et al., 2017). The highest fraction of urea lost (64%) while it broadcast on the field (Rochette et al., 2009). Over 50% of the nitrogen from urea cannot be assimilated by plants, therefore, it becomes a potential source of environmental degradation such as water pollution, eutrophication, acid rain, ammonia volatilization loss, nitrous oxide emission, global warming and stratospheric ozone depletion (Sutton et al., 2011; Puga et al., 2020). Surface application of granular urea promotes significant gaseous loss causing of low NUE (Lichiheb et al., 2019) and ultimately lower N availability. However, increase of N availability and control N losses is inevitable in profitable and sustainable agriculture. Sometimes crop suffers severe N deficiency and requires urgent N recovery. In this situation, faster N mineralization is expected throughout the root zone.

Liquid urea (LU) had been recommended to use for the warm periods during a growing season for a quick recovery of N deficiency (Wesley et al., 1998). Since then, the use LU fertilizer as an alternative N source to granular urea (GU) is increasingly recommended for profitable cereal production as LU gives better performance in crop yield (Walsh et al., 2014). Walsh and Christiaens (2016) compared LU with other two other liquid N fertilizers (urea ammonium nitrate and High NRG-N) and they found that LU was the more suitable N source in terms of N uptake, NUE and yield. Liquid urea was reported to be more ecofriendly, more efficient uptake and had 19% higher NUE (McLaughlin et al., 2011) than the GU. Singh et al., 2013 found higher growth, development, N uptake along with 20% higher wheat yield in LU application than broadcast GU. Application of liquid fertilizer may be more promising in fertilizer use efficiency and lower ecological consequences compared to granular fertilizer (de Silva et al., 2017).

Faulty methods and improper scheduling of urea applications promote losses of gaseous nitrogen that lower the NUE of the GU (Zhao et al., 2015). Judicious combinations of fertilizer sources, state and fertilizer placement can attain better mineralization, efficient uptake of nutrient, economic crop production and N loss minimization. Limited studies have been reported on the comparisons between LU and GU in terms of nitrogen mineralization when the fertilizers were applied to tropical acid soils. The current study was conducted to evaluate and compare the N mineralization of LU and GU when applied to two tropical acid soils under a laboratory settings.

**Materials and Methods**

The soils used in this study were Bungor (fine, kaolinitic, isohyperthermic, typic paleudult) and Selangor (very fine, alluvium, isohypertermic, typic endoaquepts). The Bungor soil was sampled at the field station of the Faculty of Agriculture Universiti Putra Malaysia (03̊ 00́ 12.6̋ N; 101̊ 47́ 22.4̋ E) while the Selangor soil was sampled from Sungai Besar, Selangor (03̊ 42́ 20.1̋ N; 100̊ 58́ 08.0̋ E). The soils were sampled from the top 15 cm depth and were air-dried, ground and sieved through a 2.00 mm sieve. The soils were analysed for total N using the dry combustion method on a LECO’s TruMac (LECO, 2018) CNS analyser, cation exchange capacity (CEC) using the leaching method (Chapman, 1965), soil particle distribution using the pipette method (Teh and Talib, 2006), the gravimetric water content was determined at field capacity (Tan, 2005), and pH was determined in 1:2.5 (soil: water) ratio and measured using Metrohm827 pH meter. The properties of both soils are shown in Table 1. The LU and GU were applied to the two soils at either 0, 300, 400 or 500 mg/kg soil to determine N mineralization during four weeks of incubation. Both GU and LU fertilizer were applied to the soil surface. Freshly prepared 1.20% (w/v), 1.60% (w/v) and 2.00% (w/v) of urea solution @ 2.5 mL/100g soil were applied for 300, 400 or 500 mg/kg soil respectively. The N mineralization was analysed weekly following destructive technique (Keeney and Nelson, 1982). In this method, 20 gm soil was extracted with 40 mL of potassium chloride - phenyl mercuric acetate(KCl-PMA) solution that was distilled with micro-Kjeldhal steam distillation unit and titrated against 0.01N HCl solution. The urea-N was estimated by extracting 20 gm soil with 40 mL of KCl-PMA solution. The solution was reacted with colouring reagents (Di-acetyl monoxime and Thio-semi-carbazide) and the colour intensity was measured with calibrated spectrophotometer (CECIL, CE1011) at 528 nm wavelength (Douglas and Bremner, 1970).The experiment was conducted as a complete factorial design and the experimental units were arranged in a completely randomized design with four replicates.

**Statistical analysis**

Data were statistically analysed as a complete factorial experimental design with Statistical Analysis Software (SAS) 9.4 (SAS, 2018) and differences between treatment means were compared using the least significant difference (LSD) test at the 5% level of significance.

**Results**

**NH4+-N and NO3--N concentrations**

At the same application rate, higher NH4+-N and NO3--N concentrations were recorded in LU treated soils compared to the GU from both soil series. The NH4+-N concentrations decreased while NO3**-**-N concentrations increased with increasing application rates and incubation time. Among the two soil series, NH4+-N concentrations were higher and NO3--N were lower in Bungor soil series than the Selangor soil series when compared at the same rate of LU and GU applications. The NH4+-N concentrations were high in the first week and then decreased gradually to the fourth week in Bungor soil series but in Selangor soil series, it was also high in the first week but drastically lower beginning from the second week of incubation. Similar patterns were recorded from both LU and GU treated soils. On the other hand, the NO3--N concentrations were low in the first week and it increased gradually to the fourth week in Selangor soil series but in Bungor soil, it was also low in first week but rapidly increased up to the third week and later started lowering. Similar observations were recorded in both LU and GU treated soils.

In Bungor soil series, the amount of NH4+-N concentration and rate of urea mineralization (%) in the soils were highest in the first week of incubation and then gradually decreased until the fourth week of incubation, both for LU and GU treatments (Figure 1A). In GU400 treated soils, 45.51%, 29.09%, 18.35% and 10.26% of urea mineralized to NH4+-N in the 1st, 2nd, 3rd and 4th week of incubation respectively, which were the highest among the GU treatments in respective weeks. The mineralization rates increased with GU application rate. In LU400 treated soils, 52.70%, 31.60%, 15.46% and 9.08% of urea were mineralized to NH4+-N in the 1st, 2nd, 3rd and 4th week of incubation respectively and similar to the GU, the mineralization rates increased with LU application rates. In the first two weeks of incubation, the mineralization rates were higher in LU than GU but in the last two weeks, the mineralization rates were higher in GU than LU treated soils. The lowest concentration and rate of urea mineralization (%) to NO3--N in the Bungor soils were recorded in the first week and the values increased gradually until the third week of incubation and then the values decreased at the fourth week of incubation (Figure 2A). The same patterns were observed in both GU and LU treated soils. The highest rate of NO3--N mineralization (%) in the GU treated soils was highest in the GU300 when compared at the same week of incubation. Similarly, the highest rate (%) of NO3 --N in the LU treated soils was found in the LU300soils. The rate of NO3--N mineralization (%) was higher in the LU than GU treated soils when compared at the same incubation week and application dosage.

In Selangor soil series, the amount of NH4+-N concentration and rate of urea mineralization to NH4+-N were highest in the first week of incubation for both LU and GU treated soils, and the values decreased gradually until the fourth week of incubation (Figure 1B). In GU500 treated soils, 22.41%, 6.62%, 4.04% and 2.50% of urea mineralized to NH4+-N in 1st, 2nd, 3rd and 4th week of incubation respectively, which were the highest among the GU treatments in respective weeks. The mineralization rates increased with GU application rate. Similarly, the LU500 treated soils mineralized 31.98%, 7.50%, 4.44% and 2.85% of urea to NH4+-N in the 1st, 2nd, 3rd and 4th week of incubation respectively, which were the highest among the LU treatments in respective weeks and the rate of urea mineralization to NH4+-N increased with the LU application rate similar to what was observed on the GU treated soils. However, the concentration and rate (%) of urea conversion to NO3--N in Selangor soil series were lowest in the first week of incubation and then increased gradually until the fourth week of incubation (Figure 2B). The highest rate of urea conversion to NO3--N among the GU and LU treated soils were recorded in the GU300 and LU300 soils, respectively. When compared at the same application rate and incubation week, the rate of urea conversion to NO3--N in the LU treated soils were higher than the GU treated soils.

**Urea-N remaining (%) in the soil**

The amount of urea-N remaining in both soils decreased with time with higher amount of remaining urea-N recorded in the GU than the LU treated soils when compared at the same application rate. The urea-N concentrations in both soil were higher in soils receiving higher rates of urea application. The amounts of urea-N remaining both soil were high in the first week of incubation and gradually decreased up to the second week after which no more urea-N was detected. The higher amount of urea-N was remained in GU than LU treatments meaning that NH4+-N and of NO3--N conversion was the slower along with total NH3 loss was higher in GU treated soils. The urea-N remained was highest in the first week for all the treated soils whose were continued to the second week and no urea-N was present in GU and LU treated soils after the second week of incubation in both soil. In Bungor soil series, the amounts of urea-N remaining in the soil were the highest in the first week, and then decreased gradually from the second week of incubation in LU and GU treated soils (Figure 3A). However, the percentages of urea-N remaining in the soils treated with higher GU rates were higher than those with lower application rates in the first and second week of incubation and after third week of incubation, no urea-N was detected in any of the treated soils. In LU treated soils, the percentages of urea-N remaining was higher in the first week and lower in the second week but equal in application rates at each week and after third week of incubation, no urea-N was detected like GU treated soils. In Selangor soil series, the amounts of urea-N remaining in the soil were the highest in the first week, and then decreased gradually from the second week of incubation in LU and GU treated soils (Figure 3B). The percentages of urea-N remaining in the soils treated with higher GU rates were higher than those with lower application rates in the first and second week of incubation and after that, no urea-N was detected in any of the treated soils. The percentages of urea-N remaining in the soils treated with higher LU rates were higher than those with lower application rates in the first week but equal in application rates in the second week of incubation and after third week of incubation, no urea-N was detected like GU treated soils. The amount of urea-N remaining in Selangor soils were significantly (P<0.05) higher than that of Bungor during the first and second week of incubation when compared at the same application rate for both GU and LU treatments.

**Discussion**

Immediately after application, urea granules absorb water and dissolve when it comes into contact with the soil and consequently hydrolysis process which is driven by urease enzymes will release NH4+ and HCO3- (Mariano et al., 2019) and subsequently NH3 gas will be released (Mariano et al., 2019). Soil moisture content is one of the influential factor which starts urea hydrolysis process (Abera et al., 2012). But in case of LU, the process of urea hydrolysis started earlier than GU treated soils because the urea had already been hydrolysed even before it was applied to soil while the GU needed time to absorb water before it can be hydrolysed. It has been reported that the highest percentage of applied urea will be hydrolysed within the first four days of application and LU mineralization to NH4+-N and of NO3--N was the faster than GU treatments. Only the LU application could ensure the uniform distribution of urea-N in the soil column. It can increase the adsorption of NH4+-Nby the soil particles, which prevent further conversion to NH3 gas (Rochette et al., 2013).

The mineralization process influenced the amount of urea-N remaining in the soils. The faster nitrification and higher accumulation of NH4+ decreased urea-N remaining from the applied urea in the soil (Junejo et al*.,* 2011). The LU which was in aqueous form had faster N mineralization and more NH4+ accumulation in the soils compared to the GU and therefore the LU had lower urea-N remaining in the soils.

 The concentration of NH4+-N was lower in Selangor for both the GU and LU treatments because the clayey Selangor soil series (Incepti*sols*) may had adsorbed more NH4+-N than the more sandy Bungor soil series (Ulti*sols*). On the other hand, NO3--N concentration was higher in the Selangor soil than the Bungor soil because the former had higher CEC (16.48 cmol+/kg) than the former (5.78 cmol+/kg). The Selangor soils would repel the negatively charged anionic NO3- and therefore the NO3- concentration was higher in the Selangor soils. This statement was supported by Mukhopadhyay et al*.* (2019). The NH3 volatilization loss was lower in Selangor soil for both GU and LU since most NH4+ was adsorbed by the soil particles, hence less amount of NH4+ was available to be converted to NH3. Based on the results of this study, the LU was more efficient than the GU because soils treated with LU had higher concentrations of NH4+-N and NO3--N which were distributed throughout the soil profile. Most of the NH4+-N was adsorbed by the soil particles, hence reducing further possible losses. The LU might have a greater NUE than the GU in soils since the total N loss was lower in the LU treated soils. However this needs to be further confirmed by a field study.

**Conclusions**

The results suggest that the LU would be a better source of urea N fertilizer as it resulted in higher NH4+-N and NO3--N mineralization in the soils compared to the GU. In addition, the LU performed better in the sandy clay loam Bungor soils than the clayer Selangor soils because the former had higher concentrations of available NH4+-N and NO3--N. Therefore, the use of LU would benefit the farmers because it would have higher NUE than the GU. However, the results should be confirmed by glasshouse pot experiments and field trials.

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**Author contributions**

Significant contributions were paid by each author for this publication. All authors collectively design this study, first author executed the study, collected and analysed data statistically, wrote first draft of the article. All the authors contributed to the finishing version of the manuscript and have approved to publish.

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**Table 1.** Physico-chemical properties of the soils

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| Soil properties | Bungor soil | Selangor soil |
| Texture analysis | 28.44% clay, 2.28% silt and 69.28% sand | 68.11% clay, 29.85% silt and 2.04% sand |
| USDA soil texture class | Sandy clay loam\* | Clay |
| Moisture content at field capacity (%)  | 23.74 | 27.89 |
| pH | 4.93 | 6.03 |
| Total C (%) | 1.41 | 3.46 |
| Total N (%) | 0.07 | 0.27 |
| NH4+- N (mg/kg) | 16.31 | 10.07 |
| NO3-- N (mg/kg) | 11.41 | 27.11 |
| CEC (cmol+/kg) | 5.78 | 16.48 |

\*(Min, 1967)

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**Figure 1.** NH4+-N concentration in Bungor (A) and Selangor(B) soil treated with different rate of LU and GU fertilizers during the four weeks of incubation. Vertical bars on the graphs show the standard errors.

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**Figure 2.** NO3--N concentration in Bungor (A) and Selangor(B) soil treated with different rate of LU and GU fertilizers during the four weeks of incubation. Vertical bars on the graphs show the standard errors.

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**Figure 3.** Urea-N remaining (%) in Bungor (A) and Selangor(B) soil treated with different with different rate of LU and GU fertilizers during the four weeks of incubation. Vertical bars on the graphs show the standard errors.