COMPARATIVE EFFICIENCY OF LIQUID AND GRANULAR UREA FERTILIZERS ON NITROGEN DYNAMICS, GROWTH PERFORMANCE AND YIELD OF GRAIN CORN (*Zea mays* L.)

By

MD. MOTASIM AHMMED

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Doctor of Philosophy

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DEDICATION

To my respected parents,

To my beloved wife and also

To my lovely children

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Doctor of Philosophy

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Urea is the most popular, user-friendly nitrogen (N) fertilizer having significant contributions to worldwide crop production. Urea mineralization regulates the N dynamics and N content of the soil. A huge amount of N from granular urea (GU) might be lost through nitrate (NO_3) leaching, ammonia (NH_3) volatilization, and denitrification resulting in low Nitrogen Use Efficiency (NUE). It was hypothesized that the N losses could be minimized if urea hydrolysis is fast so that the ammonium cations (NH_4^+) are more uniformly distributed throughout the soil profile. There is no single method which can effectively restrict NO_3 leaching and NH_3 and N_2O gaseous losses from applied urea except by reducing the application rate. The general objective of the study was to compare the effectiveness of GU and liquid urea (LU) with its split application as N sources for grain corn (*Zea mays* L.) grown cultivation. The first experiment was an incubation study conducted in laboratory settings to compare the N dynamics and losses in two soil series incubated with either LU or GU at 0, 300, 400 or 500 mg/kg of soil arranged in a Completely Randomized Design (CRD) layout with four replicates. The NH_4^+ and NO_3^- concentrations in the soils were measured for four weeks, while the N leaching loss was measured for ten pore volumes. Ammonia volatilization, N_2O emission, and soil N content were measured throughout the 30 days incubation period. The results showed that higher NH_4^+ and NO_3^- concentrations at the same application rate were detected in the LU applied soils. The leaching loss of N was higher in GU treated soils than that of LU, and it increased with an increase in urea application rate. The N_2O emission and NH_3 volatilization loss were also higher in GU than in the LU treated soils. The total N content that remained in the LU treated soils after incubation was higher than that of GU. The second experiment was conducted in a glasshouse to evaluate the effects of LU application frequency on the growth, yield and NUE of grain corn. The treatments consisted of a control (no urea application), granular urea applied as two equal splits (GU2S), liquid urea applied as two equal splits (LU2S), liquid urea applied as three equal splits (LU3S) and liquid urea applied as four equal splits (LU4S) arranged in a Randomized Completely Block Design (RCBD) with four replicates.

Results indicated that among all the treatments, the grain corn receiving LU3S had the highest stem diameter (2.03 cm), cob weight (248.25 g), 100-grain weight (29.08 g), N uptake (133.96 kg/ha) and NUE (72.84%). The highest grain yield was recorded in the LU3S (6766.77 kg/ha) treatment, which was 37% higher than the GU2S (4942.90 kg/ha) treatment. The third experiment was also a glass house study conducted to determine the effects of application method and rates of LU on grain corn production. The experiment was conducted as an RCBD with four replicates. The fertilizer treatments were U0 = Control, GU100 (GU applied 100% in two splits at 10th and 28th DAS), LU100 (LU applied 100% a in two splits at 10th and 28th DAS), LU50 (LU applied 50% in two splits at 10th and 28th DAS) and LU33 (LU applied 33% in two splits at10th and 28th DAS). The higher ear length (22.88 cm), 100-grain weight (22.50 g), grain weight (102.17 g/plant), husk weights (25.26 g/plant) and grain yield (6249.03 kg/ha) were observed in the LU100 treatment, but the values were statistically similar to GU100 and LU50 treatments. The N uptake (%) by the grain was also highest in LU100 (1.489 g/plant), while the value in LU50 (1.272 g/plant) was higher than the GU100 (1.159 g/plant), and the highest N uptake was recorded in LU100 (102.83 kg/ha). The NUE was highest in LU50 (66.92%), followed by LU100 (51.47 %) treatment. The final experiment was a two-cycle field study conducted to evaluate the N uptake and NUE of different LU application splits, along with their effects on the growth, development and yield of grain corn in the field and the experiments were conducted as a RCBD with four replicates. The treatments were T0 (No urea) as a control, T1 (100% GU in two splits at 10th and 28th DAS), T2 (100% LU in two splits at 10th and 28th DAS), T3 (100% LU in three splits 10, 40 and 65 DAS), T4 (50% LU in two splits at 10th and 28th DAS) and T5 (50% LU in three splits 10, 40 and 65 DAS). Results revealed that the ear length, cob diameter, cob weight, number of seeds per kernel row, grain weight /plant, 100-grain weight and grain yield were recorded highest in T3 for both seasons. However, the 100-grain weight of T3 (50.51 g) was not statistically different from T2 (49.02 g). The highest grain yield was also recorded in T3 (11019.23 kg/ha), followed by the T2 (9902.51 kg/ha) treatment. The grain yield data between the two seasons were similar when compared within similar treatment. The N uptake (161.92 kg/ha) was highest in T3, but there was no statistical difference in NUE between T3 (77.42 %) and T5 (75.42 %). Overall, the study suggests that the LU was a better N fertilizer source than GU because N mineralization and N availability were more rapid while N leaching and gaseous losses were significantly lower. The results also suggest that T3 (100% LU applied in 3 splits) was the most efficient treatment for optimizing the growth and yield of grain corn.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

PERBANDINGAN KECEKAPAN BAJA UREA CECAIR DAN BUTIRAN PADA DINAMIK NITROGEN, PRESTASI PERTUMBUHAN DAN HASIL JAGUNG BIJIRAN (*Zea mays* L.)

Oleh

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Urea ialah baja nitrogen (N) yang paling popular dan mesra pengguna yang mempunyai sumbangan besar kepada pengeluaran tanaman di seluruh dunia. Mineralisasi urea mengawal dinamik N dan kandungan N tanah. Sebilangan besar N daripada urea berbutir (GU) mungkin hilang melalui larut lesap nitrat (NO_3), pemeruapan ammonia (NH_3), dan penyahhidratan yang mengakibatkan Kecekapan Penggunaan Nitrogen (NUE) yang rendah. Telah dihipotesiskan bahawa kehilangan N boleh diminimakan jika hidrolisis urea adalah cepat supaya kation ammonium (NH_4^+) disebarkan dengan lebih seragam ke seluruh profil tanah. Tiada kaedah tunggal yang berkesan bagi menyekat larut lesap NO_3 dan kehilangan gas NH_3 dan N_2O daripada penggunaan urea kecuali dengan mengurangkan kadar penggunaan. Objektif umum kajian ini adalah untuk membandingkan keberkesanan GU dan urea cecair (LU) dengan aplikasi berpecahnya sebagai sumber N untuk penanaman jagung bijian (*Zea mays* L.). Eksperimen pertama ialah kajian pengeraman yang dijalankan dalam persekitaran makmal untuk membandingkan dinamik dan kehilangan N dalam dua siri tanah yang diinkubasi dengan sama ada LU atau GU pada 0, 300, 400 atau 500 mg/kg tanah yang disusun dalam Reka Bentuk Rawak Lengkap (CRD) dengan empat replika. Kepekatan NH_4^+ dan NO_3^- dalam tanah diukur selama empat minggu, manakala kehilangan larut lesap N diukur untuk sepuluh isipadu liang. Pengwapan ammonia, pelepasan N_2O, dan kandungan N tanah diukur sepanjang tempoh 30 hari eraman. Keputusan menunjukkan bahawa kepekatan NH_4^+ dan NO_3^- lebih tinggi pada kadar penggunaan yang sama telah dikesan dalam tanah yang diberikan LU. Kehilangan larut lesap N adalah lebih tinggi dalam tanah yang dirawat GU berbanding LU, dan ia meningkat dengan peningkatan kadar penggunaan urea. Pelepasan N_2O dan kehilangan volatilisasi NH_3 juga lebih tinggi dalam GU berbanding tanah yang dirawat LU. Jumlah kandungan N yang kekal dalam tanah dirawat LU selepas pengeraman adalah lebih tinggi daripada GU. Eksperimen kedua telah dijalankan di rumah kaca untuk menilai kesan kekerapan penggunaan LU terhadap pertumbuhan, hasil dan NUE jagung bijirin. Rawatan terdiri daripada kawalan (tiada penggunaan urea), urea berbutir digunakan sebagai dua pecahan sama (GU2S), urea cecair digunakan sebagai dua pecahan sama (LU2S), urea cecair digunakan sebagai tiga pecahan sama (LU3S) dan urea cecair digunakan sebagai empat pecahan sama (LU4S) disusun dalam Reka Bentuk Blok Rawak Lengkap (RCBD) dengan empat replika. Keputusan menunjukkan bahawa antara semua rawatan, jagung bijirin yang menerima LU3S mempunyai nilai tertinggi bagi diameter batang (2.03 cm), berat tongkol (248.25 g), berat 100-butir (29.08 g), serapan N (133.96 kg/ha) dan NUE (72.84%). Hasil bijirin tertinggi dicatatkan dalam rawatan LU3S (6766.77 kg/ha), iaitu 37% lebih tinggi daripada rawatan GU2S (4942.90 kg/ha). Eksperimen ketiga juga merupakan kajian rumah kaca yang dijalankan untuk menentukan kesan kaedah aplikasi dan kadar LU terhadap pengeluaran jagung bijirin. Eksperimen telah dijalankan sebagai RCBD dengan empat ulangan. Rawatan baja adalah U0 = Kawalan, GU100 (GU menggunakan 100% dalam dua pecahan pada DAS ke-10 dan ke-28), LU100 (LU menggunakan 100% a dalam dua pecahan pada DAS ke-10 dan ke-28), LU50 (LU menggunakan 50% dalam dua pecahan pada DAS ke-10 dan ke-28) dan LU33 (LU menggunakan 33% dalam dua pecahan pada DAS ke-10 dan ke-28). Nilai panjang telinga (22.88 cm), berat 100 biji (22.50 g), berat bijian (102.17 g/pokok), berat sekam (25.26 g/pokok) dan hasil bijirin (6249.03 kg/ha) diperhatikan lebih tinggi dalam rawatan LU100 , tetapi nilainya secara statistik serupa dengan rawatan GU100 dan LU50. Serapan N (%) oleh bijirin juga tertinggi dalam LU100 (1.489 g/pokok), manakala nilai tersebut dalam LU50 (1.272 g/pokok) adalah lebih tinggi daripada GU100 (1.159 g/pokok), dan serapan N tertinggi direkodkan adalah pada LU100 (102.83 kg/ha). NUE tertinggi adalah pada LU50 (66.92%), diikuti oleh rawatan LU100 (51.47%). Eksperimen terakhir ialah kajian lapangan dua kitaran yang dijalankan untuk menilai pengambilan N dan NUE bagi pecahan aplikasi LU yang berbeza, bersama dengan kesannya terhadap pertumbuhan, perkembangan dan hasil jagung bijirin di ladang dan eksperimen dijalankan sebagai RCBD dengan empat ulangan. Rawatan adalah T0 (Tiada urea) sebagai kawalan, T1 (100% GU dalam dua pecahan pada DAS ke-10 dan ke-28), T2 (100% LU dalam dua pecahan pada DAS ke-10 dan ke-28), T3 (100% LU dalam tiga pecahan 10, 40 dan 65 DAS), T4 (50% LU dalam dua pecahan pada DAS ke-10 dan ke-28) dan T5 (50% LU dalam tiga pecahan 10, 40 dan 65 DAS). Keputusan menunjukkan bahawa panjang telinga, diameter tongkol, berat tongkol, bilangan biji setiap baris isirong, berat biji/tanaman, berat 100 biji dan hasil bijian dicatatkan tertinggi dalam T3 untuk kedua-dua musim. Walau bagaimanapun, berat 100 biji T3 (50.51 g) tidak berbeza secara statistik daripada T2 (49.02 g). Hasil bijirin tertinggi juga dicatatkan dalam T3 (11019.23 kg/ha), diikuti oleh rawatan T2 (9902.51 kg/ha). Data hasil bijirin antara kedua musim adalah serupa jika dibandingkan dalam rawatan yang serupa. Serapan N (161.92 kg/ha) adalah tertinggi dalam T3, tetapi tiada perbezaan statistik dalam NUE antara T3 (77.42 %) dan T5 (75.42 %). Secara keseluruhan, kajian menunjukkan bahawa LU adalah sumber baja N yang lebih baik daripada GU kerana mineralisasi dan ketersediaan N adalah lebih cepat manakala N yang lesap dan kehilangan gas adalah jauh lebih rendah. Keputusan juga menunjukkan bahawa T3 (100% LU digunakan dalam 3 pecahan) adalah rawatan yang paling berkesan untuk mengoptimumkan pertumbuhan dan hasil jagung bijirin.

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This thesis was submitted to the Senate of the Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Doctor of Philosophy. The members of the Supervisory Committee were as follows:

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This is to confirm that:

- the research conducted and the writing of this thesis was under our supervision;
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AAS	Atomic absorption spectrometry
Al	Aluminium
ANOVA	Analysis of variance
C	Carbon
°C	Degree Celcius
Ca	Calcium
CEC	Cation exchange capacity
CH_4	Methen
CO_2	Carbon dioxide
CNS	Carbon nitrogen and sulpher
DAM	Di-acetyl monoxime
DAS	Days after sowing
FAO	Food and agriculture organization
GHG	Greenhouse gas
HNRGN	High NRG-N
GU	Granular urea
HP	Hewlett-Packard
H4OAC	Ammonium oxy-acetate
IPCC	Intergovernmental panel on climate change
IPNI	International plant nutrition Institute
K	Potassium
KCL	Potassium Chloride
K2SO4	Potassium Sulphate
L	Litre
LU	Liquid urea

M	Molar
MARDI	Malaysian Agricultural Research and Development Institute
Mg	Magnesium
mL	Millilitre
MoP	Muriate of Potash
N	Nitrogen
N_2O	Nitrous oxide
NUE	Nitrogen use efficiency
NH_3	Ammonia
NH_4	Ammonium
NO	Nitrite
NO_3	Nitrate
OM	Organic matter
P	Phosphorus
PMA	Phenyl mercuric acetate
RCBD	Randomized completely block design
SAS	Statistical analysis system
SOM	Soil organic matter
SPAD	Silicon photon activated diode
TSC	Thio-semicarbazide
TSP	Triple super phosphate
UAN	Urea ammonium nitrate
USG	Urea super granule
USA	United states of America
USDA	United states department of agriculture

CHAPTER I

INTRODUCTION

1.1 Background of the study

Corn (*Zea mays* L.) is one of the most important and extensively cultivated cereal crops all over the globe. It is used as food, animal feed and manufacturing goods such as milk to toothpaste, shoe polish, ethanol in the first world, provides food, feed and dietary safety in the third world's nations of Africa, Asia and Latin America (Ranum et al., 2014; USAGov, 2019). It is grown on about 160 million ha of land, 11% area of the entire world's cropland (Linquist et al., 2012), and above 73% of the corn cultivated area is located in developing countries. It is expected that the demand for corn for food and feed will be rising, governed by higher population growth and commercial advancement (Shiferaw et al., 2011).

In Malaysia, the contribution of the agriculture sector was 7.4% of GDP in 2020 (Hirschmann, 2021; USDA, 2022) and the production of palm oil was 19000 thousand metric tons (TMT), rice 1780 TMT, corn 60 TMT and peanut 2 TMT in the year 2021-22 (USDA, 2022). The corn grown is mainly sweet corn as there is no commercial grain corn farm (Nor et al., 2019) that needs to import cent per cent of its demand (Anim, 2017).

Urea is the most widespread and user-friendly, as well as the most significant nitrogen (N) fertilization source globally (Kira et al., 2019). It covers 73.4% of all N fertilizer use in the world (Sutton et al., 2011; Heffer & Prud'homme, 2016). But the frightening problem related to the urea fertilizer is its huge nitrogen loss (20% to 60%), low nitrogen use efficiency (NUE) (30 - 40%) while it was reported to be 10 - 50% in corn (Hirel et al., 2011; McAllister et al., 2012). The highest fraction of urea loss (64%) was found when it was broadcasted on the field (Rochette et al., 2009). Nitrogen loss to the environment from the applied urea is significantly greater during the first 30–50 days of application as nitrous oxide (N_2O) and ammonia (NH_3) emissions (Zhao et al., 2015). More than 50% of the nitrogen from urea is not assimilated into plants, and this can be a probable cause of ecological degradation such as water pollution, eutrophication, acid rain, global warming, and stratospheric ozone depletion, which is an alarming concern. Nitrate leaching, NH_3, and N_2O gaseous emissions, which result in low NUE in the urea fertilizers can pollute the environment (Sutton et al., 2011). About 30% of urea was missing from the surface application (Zhang et al., 2010).

Among all plant nutrients, N is a major and crucial one controlling plant development and agriculture production systems required in a massive amount by plants (Sadras & Lawson, 2013). It is an essential plant nutrient and plays a vital role in increasing crop production, confirming food security, and applying a huge amount of chemical N fertilizers (Mueller et al., 2012; Shibata et al., 2017). In corn production, N is most important (Demari et al., 2016), and contributes 30–50% to corn yield increase globally (Heffer, 2009).

Nitrogen loss as NH_3 volatilization is the most problematic. This loss becomes more alarming when urea is broadcasted (Mira et al., 2017). These losses in farming areas range as high as 64% of applied N, depending on the variations of sources, rates, places, and times of N fertilizers applied (Pan et al., 2016). Ammonia volatilization is a major process of N loss from urea (Zhu & Wen, 1992), badly polluting ecosystem and man-animal health separately and or simultaneously. By addressing these losses, it may be possible to increase the NUE (Ni et al., 2014). The amount of NH_3-N losses are affected by soil pH, buffer capacity, cation exchange capacity (CEC), soil organic matter (SOM), along with N source, N dose (Rimski-Korsakov et al., 2012), fertilization time, and placement in the corn field (Li et al., 2021; Randall & Sawer, 2008).

Agricultural soil is a prime source of N_2O emission that is active for lowering the NUE of applied urea (Zhang et al., 2019; Yao et al., 2020), the most injurious greenhouse gas (IPCC, 2014). Microbial nitrification and denitrification processes in general soils contribute about 70% of the world N_2O emissions (Braker & Conrad, 2011; Syakila & Kroeze, 2011). The denitrification is a major contributor to overall N_2O emissions from applied urea while soil having relatively higher moisture content and low (ranging from 5.0 to 7.5) soil pH (Clark et al., 2012; Goulding, 2016). Regular and heavy year-round precipitation and relatively high temperatures may influence numerous biochemical processes to discharge atmospheric N_2O during N conversions (Khalil et al., 2001). The N losses increase with the increase of precipitation frequency and/or intensity along with N application rates under tropical conditions (Owino & Sigunga, 2012).

Nitrogen leaching decreases the NUE of applied N fertilizer significantly (Puga et al., 2020). Nitrogen leaching was higher in light-textured upland soil than in relatively heavier soils (Gioacchini et al., 2002), and the higher application rate of urea increased higher N leaching loss (Ma et al., 2019). On the other hand, the slower transformation of NH_4^+-N and faster hydrolysis of applied urea decreased the leaching loss of N (Zuki, 2020). When applied at the right time and placed at a proper position, N leaching from soil decreased, and the NUE was increased (Nasielski et al., 2020).

Applying N in splits according to the requirement of the corn plant can increase the grain yield as well as lowering the N losses. Corn takes up about half of its N requirement between eight leaves (V8) and tasselling (VT) stages; the duration which comprises only 30 days, while the removal of N from the soil is directly proportional with grain yield (Richie et al., 2005; Sawyer & Mallarino, 2007). Application of N at the appropriate growth stage of the crop can increase the NUE and grain productivity. Not all of the N applied to the soil cannot be absorbed by the plant, especially in areas where the rainfall is high and frequent, as the N can be lost through leaching. However, an optimum dose with proper N application timing can raise the N recovery by 58 - 70% and therefore increase the yield and quality of grain (Haile et al., 2012). The uptake of N by the corn plant after tasselling is significantly slow as the plant concentrates in translocating stocked N in the vegetative parts into grain instead of N uptake by the roots (Culman & Thomison, 2020). Nitrogen fertilizer application in three splits has been reported to effectively increase the corn's yield compared to a single application and reduced gaseous N loss significantly (Wang et al., 2016).

The use of liquid urea (LU) fertilizer is another effective technology to improve the NUE of urea fertilizer by increasing N uptake and decreasing losses from the soil. The use of LU fertilizer as an alternative to granular urea is expanding. It has been recommended for profitable cereal production because it gave a better performance in crop yield (Walsh et al., 2015), and the use of liquid fertilizer has been recommended for more economical corn production (Leikam, 2010). Liquid N fertilizers can be transported, stored and calibrated easily for a precise application. It can also be combined with other chemicals and irrigation water (Boyer et al., 2010). The LU is more ecofriendly, more efficiently taken up by crops and has 19% higher NUE than the GU (Holloway et al., 2001; McLaughlin et al., 2011). The use of LU was found to have less NH_3 loss and higher dry matter yield and increased NUE in corn than the GU (Kasim, 2009). Liquid urea can be recommended as the most suitable N source for spring wheat in terms of N uptake, NUE, and yield compared to the other two liquid (Urea ammonium nitrate - UAN and High NRG-N, a formulation with 27% N) fertilizers (Walsh & Christiaens, 2016). The appropriate rate of LU application in synchronization with the appropriate crop stage plays a vital role in maximizing corn yield, NUE and reduce the possible environmental pollution. With these views, this study was undertaken to compare the direct efficiency of LU and GU along with the effect of the splits application method of LU in grain corn production.

1.2 Problem Statement

The agricultural system is the primary source of greenhouse gas (GHG) emission and more than half of applied N to the soil as GU is lost through gaseous and leaching loss resulting in low NUE (Sutton et al., 2011; Khan et al., 2014).

The gaseous and leaching loss of N mainly depends on the rate of urea hydrolysis, N mineralization and density of urea in soil. Soil moisture content is one of the influential factors that starts the urea hydrolysis process (Abera et al., 2012) and maintained soil total N content. It affected N mineralization and losses (Fu et al., 2019). Faster hydrolysis promotes higher urea mineralization, and higher soil water content increases the hydrolysis as well as the mineralization of urea. On the other hand, fast urea hydrolysis reduced gaseous N losses as it diffused the applied urea and NH_4^+ in the deeper soil layer (Kissel, 1988). The NH_3 volatilization, N_2O emission and leaching of N increased with the increased rate of urea applications (Ma et al., 2019; Zhang et al., 2015; Degaspari et al., 2020;). However, N losses can be minimized if urea hydrolysis is fast so that the ammonium cations (NH_4^+) are more uniformly distributed throughout the soil profile. Researchers have projected several methods to reduce N losses and improve the efficiency of urea application. There is no single method that can effectively restrict NO_3

leaching, N_2O and NH_3 losses from applied urea except by reducing the application rate. The process of urea hydrolysis is started earlier in the LU application because the urea had already been hydrolysed even before it was applied to the soil. At the same time, the GU needed time to absorb water before it could be hydrolysed. Therefore, it was hypothesised that the earlier urea hydrolysis might promote faster N mineralization. The liquid urea application could be distributed throughout the soil column, ensuring lower N concentrations in the soils.

1.3 Objectives of the study

The present study was taken to evaluate the effects of LU as a source of N and its splits application to the growth parameters and yield of grain corn in a tropical environment as compared to the GU. The specific objectives were:

i. To evaluate the N mineralization and losses of liquid and granular urea on a laboratory scale.

ii. To compare NUE between LU and GU on grain corn.

iii. To determine the effects of application methods and rates of LU on grain corn.

iv. To compare the performances of LU and GU on the growth and yield of grain corn in the field.

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