UNIVERSITI PUTRA MALAYSIA

IMPROVEMENT OF NITROGEN FERTILIZER EFFICIENCY USING ENHANCED EFFICIENCY FERTILIZER UREA ON MAIZE PRODUCTION

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

June 2017

DEDICATION

"This thesis is dedicated to my late father, **Noor Affendi bin Abdul Muti**, for being the first person who believed I can make it through this journey and my mother, **Samiah bt Awang** for her endless love, support and encouragement."

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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Chairman : Professor Mohd Khanif Yusop, PhD
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The application of urea fertilizer has always been an issue due to the loss of N of up to 50% and insufficient uptake by plants. Urea when applied to soils hydrolyzes rapidly into ammonium and the subsequent accumulation of ammonium in soil results in high pH at the urea microsites which favor ammonia (NH_3) volatilization. Meanwhile, the remaining ammonium undergoes nitrification and subsequently contributes to nitrous oxide (N_2O) emissions through denitrification. Improving N fertilizer efficiency is therefore extremely crucial to minimize environmental and economic losses. Among the possible approaches is to amend urea to enhance its efficiency or by producing Efficiency Enhanced Fertilizers (EEF) by coating urea with urease and nitrification inhibitors. In this study Cu and Zn were used as urease inhibitors while 3,4-dimetylphosphate (DMPP) was used as the nitrification inhibitor. The objectives of this study were to determine the timing of N uptake by maize at different growth stages and to determine the effects of the newly developed Enhanced Efficiency Fertilizer (EEF) on N transformation, NH_3 volatilization and N_2O emission when applied to selected soil series and to determine the effects of these fertilizers on crop production and N uptake by maize in the glass house and under field conditions. Laboratory, glass house and field studies were conducted to evaluate the EEF treatments. The ^{15}N isotope was used to measure the nutrient use efficiency (NUE), nitrogen derived from fertilizer (NDFF) and nitrogen derived from soil (NDFS) using the Munchong soil series and maize as the test crop. This technique was used to determine the timing for N uptake in maize at different growth stages. The enrichment of ^{15}N in the plants was determined using emission spectrometry. The results of this study revealed that the N fertilizer uptake by the maize peaked in the 8th week after planting after which N fertilizer uptake gradually decreased. The maize was more dependent on soil N at the early stages of its growth. However, more N fertilizer was used during the active vegetative growth stage until maturity. The Nitrogen use efficiency of the maize was low, at 37% of the applied urea, and the NDFF and NDFS were 60% and 40%, respectively. In the laboratory and glasshouse studies, ten treatments of EEF were included and labeled as: urea (control); UCu (Cu coated urea); UZn (Zn coated urea); UCuZn (Cu and Zn coated urea); UDMPP (3,4-dimetylphosphate (DMPP) coated urea); UCuDMPP (Cu and DMPP coated urea); UZnDMPP (Zn and DMPP coated urea); UCuZnDMPP (Cu, Zn and DMPP coated urea); Ubio (biochar impregnated with urea); and Ug2 (geopolymer coated urea). Urea was coated with Cu, Zn and DMPP using palm stearin. Laboratory evaluation was carried out to study the N transformation, NH_3 loss and N_2O emission on the selected soil series, the Munchong and Serdang soil series. The applications of EEF treatments with micronutrients (Cu and Zn) and DMPP were effective in reducing urea hydrolysis and the nitrification process. The UCuZn resulted in 157.49 µg g^{-1} NH_4 content and 9.07% of NH_3 loss which were significantly lower than that for the control, by 54.24% and 57.76%, respectively. On the other hand, the UCuZnDMPP treatment emitted the lowest N_2O among all the treatments with the value 27.44 µg g^{-1}, 60.62% lower in comparison to the control treatment. The Serdang and Munchong soil series were used to grow the Mas Madu maize variety under glasshouse conditions. The plants were harvested after 9 weeks. The Urea (control) and EEF treatments, triple super phosphate and potassium chloride were applied as N, P and K sources at the rate of 120, 50 and 100 kg ha^{-1}, respectively, one week after direct sowing, with two applications of the EEF treatments throughout the growing period. The NH_3 and N_2O emissions were determined using closed chamber technique and the plants were harvested to measure the dry matter yield and nutrient uptake. The results of the experiment revealed that the UCuZn treatment had the highest dry matter yield, 9.31 g pot^{-1}, and lowest NH_3 volatilization, 0.68 mg-NH_3. as compared to the other treatments. The combination of EEF treatments with Cu, Zn and DMPP resulted in 35.17 µg g^{-1} of N_2O emission while the EEF treatment with either Cu, Zn or DMPP individually resulted in higher N_2O emissions, with the values 51.07, 66.99 and 46.31 µg g^{-1}, respectively. The UCuZnDMPP treatment resulted in reduction of N_2O up to 67% compared to urea (control) treatment. To evaluate the effects of the coated urea under field conditions, Mas Madu variety of maize was planted at Ladang 2, UPM, and subjected to five treatments: urea (control), UCuZn, UCuZnDMPP, Ubio and Ug2, over 9 weeks. The site was fertilized with EEF and urea (control), TSP and MOP at the rates of 120, 50 and 100 kg ha^{-1}, respectively. The plants were harvested at 63 days to measure the grain yield. The soil and plant samples were also analyzed for N, Cu and Zn content. The results showed 74.1% and 79.5% improvement in grain yield for the UCuZn and UCuZnDMPP treatments at 7547.3 kg ha^{-1} and 7688.2 kg ha^{-1}, respectively, as compared to the Urea (control) treatment which only produced 4283.6 kg ha^{-1}. It was thus concluded that the supply of N in correct amounts and at the right time are crucial to obtain maximum efficiency of the N fertilizer, which was before the 8^{th} week of planting. Further, EEF treated with Cu and Zn (urease inhibitors) and DMPP (nitrification inhibitor) can improve N use efficiency by reducing NH_3 and N_2O emission, and at the same time increase crop production.

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

PENINGKATAN KECEKAPAN BAJA N MENGGUNAKAN BAJA UREA PENINGKATKAN EFISIEN TERHADAP HASIL JAGUNG

Oleh

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Penggunaan baja urea sering menjadi suatu isu disebabkan oleh kehilangan N yang mencecah 50% menyebabkan ketidak cukupan pada pokok. Urea apabila diletakkan pada tanah, akan terhidrolisi dengan cepat disebabkan oleh pengumpulan NH_4^+ di dalam tanah dan pH yang tinggi pada laman mikro yang menyebabkan pembebasan NH_3, manakala proses nitrifikasi menyumbang kepada pembebasan N_2O. Peningkatan kecekapan baja N adalah sangat penting untuk mengurangkan kehilangan N pada persekitaran dan mengurangkan kerugian dari segi ekonomi. Salah satu caranya adalah dengan menggunakan baja EEF urea. Di dalam kajian ini, Cu dan Zn digunakan sebagai inhibitor urease dan 3,4- dimetylphosphate (DMPP) digunakan sebagai inhibitor nitrifikasi. Objektif kajian ini adalah untuk menentukan masa yang tepat bagi pokok jagung untuk mengambil N dan menentukan kesan baja EEF yang telah diubahsuai ke atas trasformasi N, penguawapan ammonia, pembebasan nitrus oksida ke atas jenis-jenis tanah yang dipilih dan untuk menetukan kesan baja-baja tersebut ke atas penghasilan tanaman dan pengambilan N dari pokok jagung. Kajian di makmal, rumah kaca dan ladang telah dilakukan untuk menilai baja-baja EEF tersebut. Teknik ^{15}N isotope telah digunakan untuk menentukan pengambilan nutrisi yang efisien (NUE), Nitrogen dari baja (NDFF) dan Nitrogen dari tanah (NDFS) menggunakan tanah Siri Munchong dan jagung sebagai tanaman. Pengayaan ^{15}N telah ditentukan menggunakan spectometri pelepasan di Agensi Nuklear Malaysia. Keputusan kajian menunjukkan pengambilan N yang paling tinggi oleh pokok jagung adalah pada minggu ke-8 selepas ditanam. Penggunaan kecekapan nitrogen oleh pokok jagung adalah rendah iaitu 37%. Tanaman jagung lebih menggunakan nutrisi nitrogen dari tanah berbanding baja di awal pertumbuhan iaitu dalam lingkungan 40% dan 60%. Di dalam kajian makmal dan rumah kaca, sepuluh rawatan EEF telah digunakan dan dilabel sebagai; urea (kawalan); UCu (Urea disaluti Cu), UZn (Urea disaluti Zn), UCuZn (Urea disaluti Cu dan Zn), UDMPP (Urea disaluti DMPP), UCuDMPP (Urea disaluti Cu dan DMPP), UZnDMPP (Urea disaluti Zn dan DMPP), UCuZnDMPP (Urea disaluti Cu, Zn dan DMPP), Ubio (Urea disaluti biochar) dan Ug2 (Urea disaluti geopolimer). Urea disaluti Cu, Zn dan DMPP adalah dengan menggunakan stearin kelapa sawit manakala urea disaluti biochar dan geopolimer disediakan oleh UTEM dan UTP. Kajian makmal telah dijalankan untuk mengkaji transformasi N, penguawapan ammonia dan pembebasan nitrus oksida pada tanah-tanah; siri Munchong dan Serdang. Penggunaan baja EEF menggunakan micronutrient (Cu dan Zn) dan DMPP adalah cara yang berkesan untuk mengurangkan hidrolisis urea dan proses nitrifikasi. Rawatan UCuZn menurunkan kandungan NH_4 sebanyak 157.49 µg g^{-1} dan 9.07% penguawapan ammonia memberikan keputusan yang signifikan berbanding Urea (kawal). Manakala rawatan UCuZnDMPP pembebasan nitrus oksida sebanyak 27.44 µg g^{-1}. Di kajian rumah kaca, tanah siri Serdang dan Munchong telah digunakan untuk menanam pokok jagung variati Mas Madu. Pokok jagung dituai selepas 9 minggu. Rawatan (kawalan) dan rawatan EEF, TSP dan MOP diletakkan sebagai sumber baja N, P dan K pada kadar 120, 50, 100 kg ha^{-1} seminggu selepas ditanam dengan dua kali aplikasi oleh rawatan EEF sepanjang tempoh penanaman. Penguawapan NH_3 dan N_2O ditentukan menggunakan teknik bekas tertutup dan tanaman dituai untuk menentukan berat kering dan pengambilan nutrient oleh pokok. Hasil dari kajian menunjukkan rawatan UCuZn memperolehi hasil kering yang paling tinggi iaitu 9.31 g $pasu^{-1}$ dan paling rendah penguapan ammonia iaitu 0.68 mg-NH_3 berbanding rawatan lain. Baja EEF dengan kombinasi Cu, Zn dan DMPP membebasan nitrus oksida sebanyak 35.17 µg g^{-1} manakala EEF disaluti Cu, Zn dan DMPP sendiri menurunkan nitrus oksida sebanyak 51.07, 66.99 dan 46.31 µg g^{-1} . Untuk menilai kesan urea bersalut di ladang, jagung Mas Madu ditanam di Ladang 2, UPM dengan lima jenis rawatan; urea (control), UCuZn, UCuZnDMPP, Ubio dan Ug2 selama 9 minggu. Ladang telah dibaja dengan EEF dan urea (kawalan), TSP dan MOP pada kadar 120, 50 dan 100kg ha^{-1} dengan menggunakan operasi pertanian yang disyorkan. Tanah dan sampel pokok dianalisis untuk kandungan N, Cu dan Zn. Kajian menunjukkan hasil sebanyak 7547.3 kg ha^{-1} and 7688.2 kg ha^{-1} pada hasil jagung bagi rawatan UCuZn dan UCuZnDMPP manakala Urea (kawalan) hanya memberikan hasil sebanyak 4283.6 kg ha^{-1}. Kesimpulannya, memberikan baja pada kadar dan masa yang tepat adalah penting bagi mendapatkan baja yang efisien iaitu pada minggu ke-8 selepas penanaman. EEF dengan disaluti Cu dan Zn (inhibitor urease) dan DMPP (inhibitor nitrifikasi) boleh meningkatkan penggunaan N dengan lebih efisien dengan mengurangkan penguapan NH_3 dan pembebasan N_2O, dan pada masa yang sama menaikkan hasil pokok.

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I certify that a Thesis Examination Committee has met on 22 June 2017 to conduct the final examination of Nur Mahfuzah bt Noor Affendi on her thesis entitled "Improvement of Nitrogen Fertilizer Efficiency Using Enhanced Efficiency Fertilizer Urea on Maize Production" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Doctor of Philosophy.

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

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LIST OF ABBREVIATIONS

EEF	Enhanced Efficiency Fertilizer
C	Carbon
Ca	Calcium
CEC	Cation exchange capacity
CO_2	Carbon dioxide
Cu	Copper
DMPP	3,4-dimethylphosphate
H^+	Hydrogen ion
H_2SO_4	Sulphuric acid
H_2O	Water
HCl	Hydrocloric acid
Ha	Hectare
MOP	Muriate of Potash
Na	Sodium
NDFF	Nitrogen derived from fertilizer
NDFS	Nitrogen derived from soil
NH_3	Ammonia
NH_4	Ammonium
NO	Nitrogen oxide
NO_2	Nitrogen dioxide
NO_2^-	Nitrite
NO_3	Nitrate
N_2	Dinitrogen
N_2O	Nitrous oxide

P	Phosphorus
TSP	triple Super Phosphate
Zn	Zinc
µg	microgram

CHAPTER 1

INTRODUCTION

Urea is one of the most important fertilizers to supply N to most crops. Nitrogen is a mobile nutrient that is easily lost from soils, therefore continuous supply of N is crucial to maintain productivity in agricultural soils. Increase in fertilizer rates can increase the yield of crops but under certain conditions the higher application rates can cause reduction in fertilizer use efficiency (Tilman et al., 2002). Urea consumption is 38% higher than other nitrogenous fertilizers available in the market due to its high concentration of N and low manufacturing cost (Nasima et al., 2011).

The main reason for increase in N fertilizer application is to increase crop production but it also results in environmental pollution (Liu et al., 2013). Research has proved that only 40 - 70% of applied nitrogen fertilizer is taken up by plants (Johnson, 2011). A significant percentage of the remaining N fertilizer ends up in the aquatic systems and atmosphere through runoff of ammonium (NH_4^+), nitrate (NO_3^-) leaching and gaseous emissions (NH_3 and N_2O) (Ju et al., 2009). For Urea or ammonium fertilizers it has been observed that 30% of surface applied N is lost to the environment (Yinsheng et al., 2010).

The National Research Council (NCR, 2002) reported that ammonia volatilization was a major concern for air quality at the regional, national and global levels. Urea is a popular solid chemical form that is used as a top-dressing-surface fertilizer. Since the late 60s, research has shown that urea is an effective topdress material based on economics and production aspects (Johnson, 2011). NH_3 is emitted 3 to 5 days after N fertilizer application and NH_3 loss results when NH_4^+ separates into gaseous NH_3 under alkaline conditions. Other factors that determine NH_3 loss from N fertilizers are soil pH and CEC (Francis et al., 2008). Soil NH_3 emission not only contributes to significant loss of N in agricultural soils but also causes acidification and eutrophication. It is also one of the indirect causes of N_2O emissions. The volatilization process involves urea hydrolysis by the urease enzyme which is produced by soil microorganisms and plant residues. During hydrolysis, the pH will be increased up to 7 and 9 in most soils and this high pH contributes to NH_3 volatilization (Liyanage, 2014).

Emission of greenhouse gases (GHG) is mostly from agricultural activities through many processes. One GHG is nitrous oxide (N_2O) which is a major non-carbon-dioxide GHG. The global warming potential of N_2O is 298 times greater than that of carbon dioxide (CO_2) in a 100 year time horizon (Forster et al., 2007). Agricultural soils contribute 3.5 Tg N_2O-N year^{-1} from the entire anthropogenic N_2O emissions (5.7 Tg N_2O-N year^{-1}). It is estimated that 70% of N_2O emission is derived from chemical N fertilizers (Ding et al., 2007a). Nitrous oxide can be emitted by nitrification, denitrification (Cavigelli and Robertson, 2001) and the reduction of nitrates to ammonium (Silver et al., 2001). While some researchers report the major source of nitrous oxide as being from nitrification under aerobic soil conditions (Shaw et al., 2006; Ma et. al., 2007), other researchers claim that it is denitrification(Zaman et al., 2008).

Concerns about NH_3 and N_2O emissions have increased and several studies had been done to develop N fertilizers which can reduce NH_3 and N_2O emissions, and increase nutrient use efficiency and uptake by plants. Enhanced Efficiency Fertilizers(EEF) have been extensively tested in this respect. Research and development on EEF have also been conducted using varied mechanisms to achieve slow release and stabilized N fertilizers that match crop's demands. These include coating of urea fertilizers to slow the dissolution rate, use of urease and nitrification inhibitors, and the use of acidifying agents to reduce the soil pH (Saggar et al., 2008; Zaman et al., 2008). Slow and controlled release urea has been widely used to delay nutrient uptake in plants after application. Slow release urea has been around since the early sixties and was first developed in the horticultural market. Subsequently, researchers focussed on coated urea to control the loss of nitrogen through leaching and volatilization (Johnson, 2011). Controlled release urea forms such as sulfur-coated urea, polymer-coated urea, urea super granules and lac-coated urea have been widely studied (Shaviv and Mikkesen, 1993). In order to improve nitrogenous efficiency, fertilizer additives were developed using chemical additives such as nitrification inhibitors, such as 2-chloro-6-trichloromethylpyridine (nitrapyrin), dicyandiamide (DCD) and 3,4-dimethypyrazole phosphate (DMPP), and urea inhibitors, such as, n-butyl-thiophospharic triamide (NBPT) and ammonium or calcium thiosulphate. The biological population in application zones can thus be reduced or chemical boundaries formed to prevent nitrogen sources from interacting with the inherent soil chemistry(Johnson, 2011).

The use of micronutrients such as Cu and Zn as inhibitors was found to reduce NH_3 loss from urea (Reddy and Sharma, 2000). Apart from being an inhibitor that controls NH_3 loss and improves N-use efficiency, micronutrient additives are also essential as crop nutrients; therefore, their use as amendments to urea could be a great option. Study by Khanif (1986) showed that a small amount of Cu can minimize NH_3 emission through urease inhibition. Volatilization of NH_3 from the soil surface can be reduced by up to 30.3% and N-use efficiency can be improved by 23.9% using urea amended with $CuSO_4$ (Reddy and Sharma, 2000). The use of micronutrients as urease inhibitors was found to be effective (Reddy and Sharma, 2000) and beneficial(Khan et al., 2015). They can retard the urea hydrolysis in soil through urease inhibition and at the same time provide essential nutrients to plants and soil. In urease-catalyzed hydrolysis of urea, the Ni (II) in the active site of urease activates the substrate and water for the hydrolysis process. The Cu and Zn act as urease inhibitors that substitute the Ni at the active site, thus inhibiting the rate of hydrolysis. Further, these materials are easily available in the market and are biodegradable (Khan et al., 2015). Coating with Cu has been tested previously to reduce ammonia volatilization and was found to result in significant reductions of up to 50% (Nasima, 2011) and further, even small amounts, can minimize ammonia volatilization through urease inhabitation (Khanif, 1986).

Another chemical that can inhibit the nitrification process is 3,4-dimethypyrazole phosphate (DMPP). DMPP is a nitrification inhibitor that deactivates the *ammonia monooxygenase* enzyme which is responsible for oxidation of $NH4^{+}$ and NO^{2-} (Dong-Gill et. al., 2012). It was found that nitrification inhibitors can reduce nitrous oxide emissions by 30-80 % (Zaman et al., 2009; Akiyama et al., 2010). The adhesive agent used to keep the nitrogen and micronutrients and DMPP together at the micro site is a natural material, palm stearin. Use of slow release nitrogenous fertilizers such as Cu and DMPP coated urea is a potential solution to increase N use efficiency by prolonged soil retention and less fertilizer application, which also results in cost efficiency (Leong, 2002).

The current study also found that adding biochar may give benefits of reducing GHG emissions such as methane (CH_4) and nitrous oxide (N_2O) and reduce ammonia loss. Biochar is a carbon (C) rich product from the pyrolysis of organic material at low temperatures (<700 °C) (Lehmann and Joseph, 2009). Another material used to coat the urea is geopolymer. Geopolymer had been studied for numerous applications to control the release of N in fertilizers. Geopolymer has compressive strength which is determined by the compactness and strength of the material with time, temperature, chemical composition, starting material and activating solution (Irfan, 2014).

There is a need to conduct a comprehensive study related to EEF with micronutrients, DMPP, biochar and geopolymer coating to improve the efficiency of urea fertilizers with low manufacturing cost. Based on this background, this study was conducted with the following objectives:

1. To determine the timing of N uptake by maize at different growth stages using the ^{15}N isotope technique.
2. To evaluate the effects of EEF treatments on N transformation, NH_3 volatilization loss and N_2O emission in selected Malaysian soils under laboratory conditions.
3. To quantify the effects of EEF on NH_3 volatilization, N_2O emission and maize yield under glass house conditions.
4. To determine the effects of EEF on plant yield and total nutrient uptake under field conditions.

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