UNIVERSITI PUTRA MALAYSIA

EFFECTS OF ZEOLITE AND LIQUID UREA ON GROWTH OF BLACK PEPPER (Piper nigrum L.)

LATIP BUNDAN

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By

LATIP ANAK BUNDAN

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

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Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Master Science

EFFECTS OF ZEOLITE AND LIQUID UREA ON GROWTH OF BLACK PEPPER (*Piper nigrum* L.)

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Chairman : Nik Muhamad Nik Ab. Majid, PhD

Faculty : Faculty of Forestry

Ammonia volatilization is one of the major problems which leads to poor urea-N use efficiency. When urea is applied to the soil, it temporarily raises soil pH and hence causes ammonia volatilization. Ammonia loss is associated with soil properties such as pH and CEC. As black pepper is known for high nutrient demand, fertilizer use efficiency and frequency need consideration in its cultivation. Thus, proper fertilizer management is essential for improving the effectiveness of fertilizers particularly N. An approach of increasing CEC that leads to improvement of soil NH_4^+ and reduction of ammonia volatilization is

promising. Therefore, this study was carried out to minimize ammonia volatilization from urea at different levels of zeolite and to improve urea-N use efficiency by mixing urea with zeolite. For the incubation study, five treatments were evaluated using close-dynamic air flow system method, arranged in a completely randomized design. Treatments evaluated were: (T0) soil alone, (T1) 2.60 g granular urea, (T2) 2.60 g liquid urea, (T3) 2.60 g liquid urea + 4 g of zeolite, (T4) 2.60 g liquid urea + 8 g of zeolite and (T5) 2.60 g liquid urea + 12 g of zeolite. At the end of this study, soil samples were analyzed for pH, exchangeable ammonium (NH_4^+) and available nitrate (NO_3^-) using standard procedures. When urea was applied in the liquid form, ammonia loss was reduced compared to granular urea but not significantly different among the treatments with and without zeolite. Besides, application of both liquid urea and zeolite was found to increase accumulation of exchangeable ammonium concentration. This could be due to retention of ammonium ions at the exchange sites of zeolite. Hence, soil column experiment under green house condition was conducted to determine the effect of treatments on growth of black pepper. The *Kuching* variety was used to evaluate the effect of these treatments on N, P and K uptake and their use efficiency including soil inorganic N (exchangeable ammonium and available nitrate), available P, exchangeable K and pH. The treatments evaluated were: (T0) no fertilizer, (T1) 6.52 g granular urea, (T2) 6.52 g liquid urea, (T3) 6.52 g liquid urea + 57.2 g of zeolite, (T4) 6.52 g liquid urea + 114.4 g of zeolite and (T5) 6.52 g liquid urea + 171.6 g of zeolite. Standard requirement for the triple superphosphate and muriate of potash were applied except for T0. The results showed that application of zeolite significantly increased soil available N, P, exchangeable K, Mg and Ca. This is because the ability of zeolite to supply essential secondary elements and to reduce soil cation loss through leaching. The highest amount of zeolite (T5) increased soil pH and it significantly improved leaf chlorophyll content, number of leaves, N, P, and K uptake and their use efficiency. Application of zeolite reduced ammonia loss and increased accumulation of available nutrients for growth of black pepper as well as improving N, P and K uptake and their use efficiency. Hence, application of zeolite could be an alternative way to minimize the use of fertilizer as well as reducing environmental pollution.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

KESAN ZEOLITE DAN UREA CECAIR PADA PERTUMBUHAN LADA HITAM (*Piper nigrum* L.)

Oleh

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Pemeruapan ammonia adalah salah satu daripada masalah utama yang mengurangkan keberkesanan urea-N. Apabila urea ditabur pada tanah, ia akan meningkatkan pH tanah secara sementara dan ini menyebabkan pemeruapan ammonia. Kehilangan ammonia adalah berkait rapat dengan sifat-sifat tanah seperti pH dan CEC. Oleh kerana lada hitam dikenali sebagai tanaman yang memerlukan nutrien yang tinggi, maka keberkesanan dan kekerapan pembajaan adalah perlu diambil kira di dalam penanamanya. Oleh itu, pengurusan baja yang sesuai adalah penting untuk memperbaiki keberkesanan baja terutamanya bagi

unsur N. Pendekatan seperti meningkatkan CEC yang menyumbang kepada peningkatan ion NH_4^+ dan mengurangkan pemeruapan ammonia adalah petanda yang baik. Oleh itu, kajian makmal telah dijalankan untuk mengurangkan kehilangan ammonia daripada urea dengan menggunakan jumlah zeolite yang berbeza dan untuk memperbaiki keberkesanan N dengan mencampur urea dengan zeolite. Untuk kajian makmal, lima rawatan telah dikaji dengan menggunakan kaedah *close-dynamic air flow system* dan disusun dengan menggunakan *completely randomized design*. Rawatan yang dikaji adalah: (T0) tanah sahaja, (T1) 2.60 g butiran urea, (T2) 2.60 g urea cecair, (T3) 2.60 g urea cecair + 4 g of zeolite, (T4) 2.60 g urea cecair + 8 g zeolite dan (T5) 2.60 g urea cecair + 12 g zeolite. Di akhir kajian, sampel tanah telah dianalisis untuk menentukan pH, NH_4^+ dan NO_3^- dengan menggunakan kaedah piawai. Apabila urea digunakan dalam bentuk cecair, kehilangan ammonia telah dapat dikurangkan jika dibandingkan dengan pengunaan butiran urea tetapi tidak signifikan diantara rawatan yang ada dan tiada zeolite. Selain itu, penggunaan urea cecair dan zeolite juga telah meningkatkan kandungan ion ammonium. Ini mungkin disebabkan oleh penahanan ion ammonium pada tempat tukaran pada zeolite. Oleh itu, kajian menggunakan kolum tanah telah dijalankan di dalam rumah hijau untuk menilai kesan rawatan pada petumbuhan lada hitam. Varieti *Kuching* telah digunakan untuk menilai kesan rawatan pada penyerapan serta keberkesanan N, P dan K termasuk kesan pada N tak organik (tukarganti ammonium dan keterdapatan nitrat), keterdapatan P, tukarganti K dan pH pada tanah. Rawatan yang dinilai adalah: (T0) tanpa baja, (T1) 6.52 g butiran urea, (T2) 6.52 g urea cecair, (T3) 6.52 g urea cecair + 57.2 g zeolite, (T4) 6.52 g urea cecair + 114.4 g zeolite dan

(T5) 6.52 g urea cecair + 171.60 g zeolite. Kadar piawai untuk triple superphosphate dan muriate of potash telah digunakan kecuali untuk T0. Keputusan telah menunjukkan penggunaan zeolite signifikan meningkatkan keterdapatan P tanah, tukarganti K, Mg dan Ca. Ini adalah disebabkan oleh kebolehan zeolite untuk membekalkan nutrien sekunder dan mengurangkan kation tanah daripada melarut lesap. Penggunaan zeolite yang terbanyak (T5) telah meningkatkan pH tanah, dan signifikan meningkatkan kandungan klorofil pada daun, jumlah daun, penyerapan dan keberkesanan N, P dan K. Penggunaan zeolite telah mengurangkan kehilangan ammonia dan meningkatkan pengumpulan ketersediaan nutrien untuk pertumbuhan lada hitam di samping memperbaiki penyerapan dan keberkesanan unsur N, P dan K. Oleh itu, penggunaan zeolite mungkin dapat dijadikan sebagai jalan altenatif untuk mengurangkan penggunaan baja sekaligus mengurangkan pencemaran alam sekitar.

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I certify that a Thesis Examination Committee has met on 9 July 2012 to conduct the final examination of Latip anak Bundan on his thesis entitled "Effects of Zeolite and Liquid Urea on Growth of Black Pepper (*Piper nigrum* L.)" 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 Master of Science.

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DECLARATION

I declare that the thesis is my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously, and is not concurrently, submitted for any other degree at Universiti Putra Malaysia or at any other institution.

LATIP ANAK BUNDAN

Date: 9 July 2012

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LIST OF ABBREVIATIONS

AAS	Atomic absorption spectrophotometry
Ca	Calcium
CEC	Cation exchange capacity
CRD	Completely randomized design
C_SCl	Caesium chloride
FIFA	Fertilizer Industry Federation of Australia
H	Hydrogen
H_2SO_4	Sulphuric acid
HCl	Hydrochloric acid
HCO_3^-	Bicarbonate
HNO_3	Nitric acid
K	Potassium
K_2SO_4	Potassium sulphate
KCl	Potassium chloride
MARDI	Malaysian Agricultural Research and Development Institute
Mg	Magnesium
mg	Milligram
MgO	Magnesium oxide
mL	Millilitre
N	Nitrogen
NaOH	Sodium hydroxide
NH_3	Ammonia
NH_4^+	Ammonium
NH_4OA_C	Ammonium acetate
NO_3^-	Nitrate
P	Phosphorus
ppm	Part per million
RCBD	Randomized complete block design
SAS	Statistical analysis system

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CHAPTER 1

INTRODUCTION

As black pepper is known for high nutrient demand, fertilizer use efficiency and frequency need consideration in its cultivation. Besides maximizing yield production, the high fertilizer use efficiency could also reduce the cost of fertilizer as well as minimizing environmental pollution. Thus, proper fertilizer management is essential for improving the effectiveness of fertilizers particularly N. Nitrogen is usually limited in soil because of loss via leaching, ammonia (NH_3) volatilization, surface runoff and denitrification (Bolan *et al*., 2004; Yan *et al*., 2003; Brady and Weil, 2002). These losses result in approximately 52% of the total N applied (Salifu *et al*., 2009; Shaviv and Mikkelsen, 1993), besides having negative impact to the environment (Edwards *et al*., 2000; Sharpley *et al*., 2000). Therefore, high amount of N fertilizer is usually applied to meet the pepper N requirement especially for *Kuching* variety (Adzemi *et al*., 1993).

Urea is considered the cheapest inorganic N source because it contains 46% N (Thompson and Meisinger, 2004). In addition, its availability and ease of handling encourage the use of urea by farmers. It is commonly broadcasted to the soil and this can reduce labour cost (Philippe *et al*., 2009). In the presence of soil water, hydrolysis occurs by urease enzyme to breakdown urea molecules into ammonium (NH_4^+) and bicarbonate (HCO_3^-) ions (Sommer *et al*., 2004). At the same time, soil pH increases because of high concentration of NH_4^+ ions exceeding the localised buffering capacity leading to ammonia volatilization (Zaman *et al*., 2009; Singh *et al*., 1994; Fan and MacKenzie, 1993; Zhengping *et al.*, 1991). Ammonia loss has been reported to be associated with soil properties, environmental conditions and method of application. In contrast to the incorporation method, major loss is associated with surface application method, a practice which leads to ammonia volatilization (Cai *et al*., 2002; Prasertsak *et al*., 2001). Under favourable conditions, ammonia loss from surface application could be up to 50% (Sommer *et al*., 2004). Therefore, these methods of application contribute to high loss of ammonia and decrease nutrient use efficiency by crops (Van der Stelt *et al*., 2005; Mohammad *et al*., 1999; Malhi *et al*., 1996).

In order to improve the efficiency of N utilization, several approaches have been used by researchers. These approaches include using urease inhibitors (Sanz-Cobena *et al*., 2011; Sanz-Cobena *et al*., 2008; Gill *et al*., 1997), soil incorporation (Nyord *et al*., 2008; Sommer *et al*., 2004), additives (Ahmed *et al*., 2010a; Latifah *et al*., 2010; Ahmed *et al*., 2006) and coating as slow-release (Ni *et al*., 2009; Liu *et al*., 2007). However, these approaches could have limitations to use especially for acidic additives such as phosphoric acid. Besides the cost involved in amending urea, corrosive characteristics require extra precautions. Therefore, additive which is highly effective and environmentally safe such as zeolite could be used to reduce ammonia loss.

Zeolites are porous minerals with pores arising from the substitution of aluminium and silicon ions (Ming and Mumpton, 1989). Zeolites have high CEC, high adsorption capacity besides having high water holding capacity (Ayan *et al*., 2005; Mumpton, 1999). In laboratory studies, ammonia volatilization from urea was significantly reduced when it was mixed with zeolite (Ahmed *et al.*, 2010a; Ahmed *et al*., 2008; Ahmed *et al.*, 2006). These studies reported that the exchange sites of zeolite increased the retention of NH_4^+ ions in the soil compared to formation of ammonia. Therefore, addition of zeolite with urea improves urea-N use efficiency (Ahmed *et al.*, 2010b; Rehakova *et al.*, 2004; Gruener *et al*., 2003).

Besides reducing ammonia volatilization, the high CEC of zeolites also reduce leaching of fertilizer cations. For example, application of zeolite to sandy soil increased the retention of NH_4^+ and directly reduced NH_4^+ ions leaching as compared to soil alone (Zwingmann *et al*., 2009). Ammonium ions tend to leach from the soil profile and move beyond the root zone when water flow is sufficient to transport the solutes (Arregui and Quemada, 2006; McNeill *et al.*, 2005). Leaching of valuable nutrients below the crop rooting zone represents economic loss and reduces nutrient use efficiency.

Reduction in ammonia loss was reported when urea was incorporated or injected in the soil (Philippe *et al*., 2009; Nyord *et al*., 2008). However, this method of application directly affects roots of plants and causes crop damage (Hanna *et al*., 2005; Stecker *et al.*, 1993; Blaylock and Cruse, 1992). Special equipment and suitable technique are also required for its application. Hence, water is required to diffuse urea into the soil layer at the time of application to reduce ammonia loss. Sanz-Cobena *et al*. (2011) reported sufficient irrigation after urea application can move urea below the soil surface and reduce ammonia volatilization. This is because urea is highly soluble in water. Clothier and Sauer (1988) found that urea hydrolysis occurred when urea penetrates into the soil profile after 1 to 2 hours of application in drip fertigation. In this way, accumulation of NH_4^+ ions does not only localize at the placement zone but diffuses to other parts of the soil, consequently reducing ammonia loss.

Amending urea with zeolite could improve soil CEC which increases the ability of soil to absorb NH_4^+ ions as well as reducing ammonia volatilization and nitrification. Applications of liquid urea which enters the soil layer could also create barrier that protects NH_4^+ ions from volatilizing. It can be hypothesized that the application of zeolite combine with liquid urea will reduce the ammonia volatilization and increase the N availability in soil. There is little knowledge about the response of black pepper when urea is applied in liquid form especially when it is mixed with zeolite. Therefore, this study was carried out to: i) minimize ammonia volatilization from urea at different levels of zeolite, and ii) improve urea-N use efficiency by mixing urea with zeolite.

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LIST OF PUBLICATION

1. Bundan, L., Nik Muhamad, A.M., Ahmed, O.H., Jiwan, M. and Kundat, F.R. (2011). Ammonia volatilization from urea at different levels of zeolite. International Journal of the Physical Sciences 6(34): 7717-7720.