



**UNIVERSITI PUTRA MALAYSIA**

***IMPROVING NITROGEN USE EFFICIENCY OF MIXED FERTILIZERS  
USING CLINOPTILOLITE ZEOLITE***

**KRYSTLE AMANDA RABAI NASIP**

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BERILMU BERBAKTI

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**MASTER OF SCIENCE  
UNIVERSITI PUTRA MALAYSIA**

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**By**

**KRYSTLE AMANDA RABAI NASIP**

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

**April 2013**

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## DEDICATION

Dedicated to my family and love ones who supported me throughout my  
research work



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

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FERTILIZERS USING CLINOPTILOLITE ZEOLITE**

By

**KRYSTLE AMANDA RABAI NASIP**

**April 2013**

**Chairman : Ahmed Osumanu Haruna, PhD**

**Faculty : Faculty of Agriculture and Food Sciences, (Bintulu)**

According to Wu and Liu (2008), up to 70% of N, 90% of P, and 70% of K are lost to the environment and not available for plant uptake. This leads to economic losses and causes serious environmental pollution. Blending mixed fertilizers with clinoptilolite zeolite where the zeolite acts as excellent carrier regulator and stabilizes mineral fertilizers could be an alternative way of formulating desired fertilizers in agriculture. An incubation experiment was carried out to compare the effects of different ratios of mixed fertilizers amended with clinoptilolite zeolite on ammonia ( $\text{NH}_3$ ) volatilization, soil exchangeable ammonium ( $\text{NH}_4^+$ ) and available nitrate ( $\text{NO}_3^-$ ) contents on a fine, loamy, siliceous, hyperthermic, *Typic Paleudults*, Ultisol (Bekenu Series). In this study, the seven treatments compared were: 14.88 g commercial fertilizer with grade 15 N:15 P:15K – zeolite (T1), 14.88 g mixed fertilizer with

grade 15 N:15 P:15 K + zeolite (T2), 22.35 g mixed fertilizer with grade 10 N:10 P:10 K + zeolite (T3), 27.87 g mixed fertilizer with grade 8 N:8 P:8 K + zeolite (T4), 40.46 g mixed fertilizer with grade 5.5 N:5.5 P:5.5 K + zeolite (T5), 2.425 g urea without additives (T6) and soil only (T7). Standard procedures were used to evaluate these treatments. Treatments with zeolite significantly reduced  $\text{NH}_3$  loss compared with urea without additives. They also improved retention of exchangeable  $\text{NH}_4^+$  and  $\text{NO}_3^-$ , and this was possible because zeolite favoured formation of  $\text{NH}_4^+$  and  $\text{NO}_3^-$  over  $\text{NH}_3$ . T1, T2, T3, T4 and T5 reduced maximum of  $\text{NH}_3$  loss from 8.4 to 0.12, 5.16, 4.02, 4.5 and 3.41% (of the N added as urea), respectively. The maximum  $\text{NH}_3$  loss occurred on day 2 for T6, day 3 for T1, day 4 for T3 and day 5 for T2, T4 and T5 of incubation. Afterwards, there was a general decline until day 15 when the  $\text{NH}_3$  loss was about 1% of the N added as urea. T1 caused the highest accumulation of exchangeable  $\text{NH}_4^+$  while T5 caused the highest accumulation of available  $\text{NO}_3^-$ . These mixed fertilizers were further tested in a pot experiment where Masmadu maize (*Zea mays* L) was used as a test crop. The objectives of this study were to evaluate the effects of the treatments on: (1) Plant height, dry matter, N, P, and K uptake N, P, and K use efficiency and their production efficiency in maize cultivation, and (2) Soil exchangeable  $\text{NH}_4^+$ , available  $\text{NO}_3^-$ , pH, exchangeable K and available P contents. The treatments evaluated were: 14.88 g commercial fertilizer with grade 15 N:15 P:15K - zeolite (T1), 14.88 g mixed fertilizer with grade 15 N:15 P:15 K + zeolite (T2), 22.35 g mixed fertilizer with grade 10 N:10 P:10 K + zeolite (T3), 27.87 g mixed fertilizer with grade 8 N:8 P:8 K + zeolite (T4), 40.46 g mixed fertilizer with grade 5.5

N:5.5 P:5.5 K + zeolite (T5) and soil only (T7). The amounts of mixed fertilizer applied were based on the standard recommendation for mature Masmadu Maize. The effect of T1 and T5 on soil total N was significant compared with T7. Treatments with higher amounts of clinoptilolite zeolite (T4 and T5) significantly improved soil total P, K and available K compared to T1 and T7. T4 and T5 caused the highest accumulation of exchangeable  $\text{NH}_4^+$  and available  $\text{NO}_3^-$  compared to T1 and T7. T4 and T5 had a similar effect on plant height, dry matter, N, P and K concentrations, and uptake and use efficiency compared with T1. It may be concluded that treatments with higher amounts clinoptilolite zeolite, 27.87 g mixed fertilizer with grade 8 N:8 P:8 K + zeolite (T4), and 40.46 g mixed fertilizer with grade 5.5 N:5.5 P:5.5 K + zeolite (T5) ensured good retention of soil exchangeable cations within the Ultisol. However, field evaluation for the treatments for at least three cropping cycles, for corn yield (cob) and cost and benefits analysis are recommended as a further research for economic viability.



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

**MEMPERTINGKATKAN KECEKAPAN PENGGUNAAN NITROGEN  
DARI BAJA CAMPURAN DENGAN MENGGUNAKAN ZEOLIT  
KLINOPTILOLIT**

Oleh

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Menurut Wu dan Liu (2008), sehingga 70% N, 90% daripada P, dan 70% daripada K hilang kepada alam sekitar dan tidak boleh digunakan untuk pengambilan tumbuhan. Ini membawa kepada kerugian dalam ekonomi dan menyebabkan pencemaran alam sekitar yang serius. Campuran baja campuran dengan zeolit klinoptilolit dimana zeolit bertindak sebagai pengatur pembawa cemerlang dan kestabilkan baja mineral merupakan kaedah alternatif dalam mengformulasi baja yang diinginkan dalam pertanian. Satu eksperimen inkubasi telah dijalankan untuk membanding kesan nisbah yang berbeza yang digabungkan dengan zeolit klinoptilolit terhadap pemeruapan ammonia ( $\text{NH}_3$ ), tukar ganti ammonium ( $\text{NH}_4^+$ ) dan kandungan nitrat ( $\text{NO}_3^-$ ) tersedia di dalam tanah yang halus, liat, silika, hyperthermic, *Typic Paleudults*, Ultisol (Siri Bekenu). Kajian ini telah membandingkan tujuh rawatan: 14.88 g baja

kompoun komersial dengan gred 15:15:15 -zeolit (T1), 14.88 g baja campuran dengan gred 15 N:15 P:15 K + zeolit (T2), 22.35 g baja campuran dengan gred 10 N:10 P:10 K + zeolit T3), 27.87 g baja campuran dengan nisbah 8 N:8 P:8 K + zeolit (T4), 40.46 g baja campuran dengan gred 5.5 N:5.5 P:5.5 K + zeolit (T5), urea 2.425 g tanpa aditif (T6) dan tanah sahaja (T7). Prosedur piawai telah digunakan untuk menilai rawatan ini. Rawatan dengan zeolit mengurangkan kehilangan  $\text{NH}_3$  dengan urea tanpa aditif. Zeolit juga telah meningkatkan keupayaan pemegang  $\text{NH}_4^+$  tukar ganti dan  $\text{NO}_3^-$  tersedia. Ini adalah berkemungkinan kerana zeolit menggalakan pembentukan  $\text{NH}_4^+$  dan  $\text{NO}_3^-$  berbanding  $\text{NH}_3$ . T1, T2, T3, T4, T5 telah berjaya mengurangkan kehilangan  $\text{NH}_3$  secara maksimum nilai 8.4 kepada 0.12, 5.16, 4.02, 4.5 and 3.41% (N yang ditambah sebagai urea) masing-masing. Kehilangan  $\text{NH}_3$  secara maksimum bagi setiap rawatan berlaku pada hari ke-2 (T6) , hari ke-3 (T1), hari ke-4 (T3) dan hari ke-5 (T2, T4 dan T5) pengeraman sebelum berlakunya penurunan umum sehingga hari ke-15 apabila kehilangan  $\text{NH}_3$  mencapai 1% daripada N yang ditambah sebagai urea. T1 telah menyebabkan pengumpulan tertinggi  $\text{NH}_4^+$  tukar ganti manakala T5 telah menyebabkan pengumpulan tertinggi  $\text{NO}_3^-$  tersedia. Baja kompoun kemudiannya diuji dengan skala yang dinaikan di dalam eksperimen menggunakan pasu di mana jagung Masmadu (*Zea mays* L) telah digunakan sebagai tanaman diuji. Objektif kajian ini adalah untuk menilai kesan rawatan ke atas: (1) Tinggi, berat kering, pengambilan N, P, dan K, kecekapan penggunaan dan kecekapan pengeluaran dalam penanaman jagung, dan (2)  $\text{NH}_4^+$  tukar ganti,  $\text{NO}_3^-$  tersedia, pH, tukar ganti K dan P tersedia. Kajian membandingkan enam rawatan: 14.88 g baja kompoun

komersial dengan gred 15:15:15 - zeolit (T1), 14.88 g baja campuran dengan gred 15 N:15 P:15 K + zeolit Zeolit (T2), 22.35 g baja campuran dengan gred 10 N:10 P:10 K + zeolit (T3), 27.87 g baja campuran dengan gred 8 N:8 P:8 K + zeolit (T4), 40.46 g baja campuran dengan gred 5.5 N:5.5 P:5.5 K + zeolit (T5) dan tanah sahaja (T7). Jumlah baja campuran yang digunakan adalah berasaskan cadangan piawai Jagung Masmadu matang. Kesan T1 dan T5 pada jumlah N-tanah adalah bererti berbanding dengan T7. Rawatan dengan jumlah zeolit klinoptilolit yang lebih tinggi (T4 dan T5) telah meningkatkan jumlah P, jumlah K dan K tersedia secara bererti berbanding T1 dan T7. T4 dan T5 menyebabkan pengumpulan tertinggi  $\text{NH}_4^+$  tukar ganti dan  $\text{NO}_3^-$  tersedia tertinggi berbanding T1 dan T7. T4 dan T5 memberi kesan yang sama pada ketinggian pokok, berat kering, kandungan N, P dan serta pengambilan kecekapannya berbanding T1. Walau bagaimanapun, kesan ini berbeza secara bererti berbanding T7. Terdapat potensi untuk baja kompaun ini digabungkan dengan zeolit untuk memberi fungsi yang lebih baik daripada baja komersial sedia ada. Dengan itu, satu kesimpulan bahawa rawatan dengan jumlah yang lebih tinggi zeolit, 27.87 g baja campuran dengan gred 8 N:8 P:8 K + zeolit (T4), dan 40.46 g baja campuran dengan gred 5.5 N:5.5 P:5.5 K + zeolit (T5) memastikan pengekalan kation tukar ganti yang baik dalam tanah dan mengurangkan kehilangan  $\text{NH}_3$ . Walau bagaimanapun, penilaian untuk rawatan sekurang-kurangnya tiga kitaran tanaman, pengukuran hasil jagung (bongkah) dan kos analisis rawatan adalah disyorkan sebagai kajian lanjutan bagi daya maju ekonomi.

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I certify that a Thesis Examination Committee has met on 18<sup>th</sup> April 2013 to conduct the final examination of **Krystle Amanda Rabai Nasip** on her thesis entitled **IMPROVING NITROGEN USE EFFICIENCY OF MIXED FERTILIZERS USING CLINOPTILOLITE ZEOLITE** 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 (Agrotechnology).

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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.



**KRYSTLE AMANDA RABAI NASIP**

Date: 18 APRIL 2013

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## LIST OF ABBREVIATIONS

N	Nitrogen
P	Phosphorus
K	Potassium
Ca	Calcium
Mg	Magnesium
Na	Sodium
Al	Aluminium
O	Oxygen
P <sub>2</sub> O <sub>5</sub>	Phosphorus pentoxide
K <sub>2</sub> O	Potassium oxide
AlO <sub>3</sub>	Aluminium oxide
SiO <sub>4</sub> <sup>-4</sup>	Silicate oxide
NH <sub>4</sub> <sup>+</sup>	Ammonium
NH <sub>3</sub>	Ammonia
NO <sub>3</sub> <sup>-</sup>	Nitrate
HPO <sub>4</sub> <sup>-2</sup>	Hydrogen phosphate
H <sub>2</sub> PO <sub>4</sub> <sup>-</sup>	Dihydrogen phosphate
HCO <sub>3</sub>	Bicarbonate
N <sub>2</sub> O	Nitrous oxide
PO <sub>4</sub> <sup>-3</sup>	Phosphate
RP	Rock phosphate

TSP	Triple superphosphate
MOP	Muriate of potash
DW	Dry weight
AAS	Atomic absorption spectrophotometry
CEC	Cation exchange capacity
DAP	Day after planting
KCl	Potassium chloride
ANOVA	Analysis of variance
mg	milligram
kg	kilogram
g	gram
$\mu\text{m}$	micrometer
M	Molar
%	Percentage
m	meter

# CHAPTER 1

## INTRODUCTION

Soil is the principal reservoir from which nutrients essential for plant growth and development are drawn (Reddy, 2004). Intensive cultivation due to high food demand has lowered soil fertility. Restoration of soil fertility is a necessity for the attainment of sustainable agriculture. Thus, fertilizer inputs are needed to cultivate crops. About 4.48 million hectares of land in 2008 were cultivated with perennial tree crops such as oil palm (Malaysia Palm Oil Board and America Palm Oil Council, 2010) where large quantities of fertilizers are required annually to sustain high crop yield. The bulk of the fertilizers used in Malaysia are for plantation crops and this accounts for 90% of all fertilizer use (Ramli *et al.*, 2012). The total import for urea, TSP and MOP since 2007 was 1,680,000 metric tonnes (Matassan, 2007). However, according to Wu and Liu (2008), up to 70% of N, 90% of P, and 70% of K are lost to the environment and not available for plant uptake. This leads to economic losses and causes serious environmental pollution.

Mixed fertilizers are the mixtures of straight fertilizer materials containing all of the three major elements and a range of minor elements (Greer, 1987). They are prepared by dry blending of straight fertilizers or by chemical reactions. Example of N:P:K grade for mixed fertilisers that have been developed such as

are 8:8:8, 13:13:22, and 15:15:15. The N:P:K indicates the percentage of N,  $P_2O_5$  and  $K_2O$ . The typical content of nutrients (N +  $P_2O_5$  +  $K_2O$ ) range from 40% to 60% (European Fertilizer Manufacturers' Association, 2000). The main advantages of using mixed fertilizers are: (i) to provide a known level of NKP, (ii) to supply all major elements, (iii) for easy application, and (iv) cost efficient comparison with one-way fertilizers (Greer, 1987).

Blending mixed fertilizers with clinoptilolite zeolite, where clinoptilolite zeolite acts as supplementary nutrients, could be an alternative way of formulating desired fertilizers in agriculture. The importance of this study is to use clinoptilolite zeolite as nutrients carrier so as to reduce  $NH_3$  loss, increase the N, P and K use efficiency of crops and to as well improve soil fertility. A slow release N fertilizer can be produced by amending urea with clinoptilolite zeolite (Omar *et al.*, 2011; Ahmed *et al.*, 2008). Urea is converted into  $NH_4^+$  by urease after which it is converted to readily leachable  $NO_3^-$  by soil bacteria (Eberl and Lai, 1992). The rate of N release is slower in three ways: (i) by sequestering urea in zeolite pores to prevent it from being leached out from the root zone; (ii) delaying the formation of ammonium ions by slowing down the release of urea with urease enzyme in soil, and (iii) by taking up ammonium ions into exchange sites and protect them from nitrification by microbes (Eberl and Lai, 1992). Zeolites containing a monovalent exchange ion such as  $NH_4^+$  (Lai and

Eberl, 1986), when mixed with P fertilizers, they take  $\text{Ca}^{2+}$  from P fertilizers thereby slowing the release of  $\text{H}_2\text{PO}_4^-$  and  $\text{NH}_4^+$  for plant uptake.

Clinoptilolite zeolite can be a mineral source of nutrients such as K, Ca, and Mg (Bagdasarov *et al.*, 2004). Clinoptilolite zeolite has three important main properties: high cation exchange capacity, high holding water capacity in its free channels, and high adsorption capacity (Bernaldi *et al.*, 2010a; Payra and Dutta, 2003) which are of great interest in agriculture. This is due to the unique feature of clinoptilolite zeolite, as it is structured in three-dimensional rigid crystalline network, formed by the tetrahedral  $\text{AlO}_4$  and  $\text{SiO}_4$ , which is made up of tunnels that are  $10^{-9}$  m in size containing internal exchange sites. These sites have high affinity for  $\text{NH}_4^+$  (Ming and Dixon, 1986; Ferguson and Pepper, 1987). These structures also ensure a permanent water reservoir in the root zone and physically protect  $\text{NH}_4$  from nitrification by microbes (Omar *et al.*, 2011; Polat *et al.*, 2004).

In various studies, clinoptilolite zeolite has been used as efficient material to increase crop yield, reducing nutrient losses and improving nutrient use efficiency (Ahmed *et al.*, 2010; Milosevic and Milosevic, 2009; Oosterhuis and Howard, 2008). In Bernardi *et al.* (2010b) study, urea amended with clinoptilolite zeolite improved the efficiency of N use where the effects of urea



and zeolite (32.6%) were statistically equivalent to treatments with urease inhibitor (37%). Ahmed *et al.* (2010) also reported that in maize plant, N, P, and K use efficiency were significantly affected by the combination of a chemical fertilizer and clinoptilolite zeolite. In the same study, application of lowest dosage of chemical fertilizer and highest dosage of clinoptilolite zeolite gave the highest N, P, and K concentrations in soils. In another report, application of the highest dosage of chemical fertilizer and highest dosage of clinoptilolite zeolite improved plant height, diameter and yield of sugarcane, and the treatment was considered profitable (Junrungreang *et al.*, 2002).

There is still a dearth of information about the effectiveness of mixed fertilizers and clinoptilolite zeolites in minimizing  $\text{NH}_3$  volatilization and as well as improving soil chemical properties and increasing crop height, dry matter and N, P and K use efficiency. The objectives of this study were to: (i) compare the effect of different ratios of mixed fertilizer amended with zeolite on  $\text{NH}_3$  volatilization, soil exchangeable  $\text{NH}_4^+$ , and available  $\text{NO}_3^-$  contents of an acid soil, and (ii) clinoptilolite zeolite to improve selected soil chemical properties, nutrient uptake, and nutrient use efficiency using maize as a test crop. In this study, it was expected that the right proportion of mixed fertilizers and clinoptilolite zeolite will increase N use efficiency and improve crop yield.

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## LIST OF PUBLICATIONS

1. Rabai. K. A., O. H. Ahmed and S. Kasim, 2012. Improving formulated nitrogen, phosphorus and potassium compound fertilizer using zeolite. *African Journal of Biotechnology*. **11**(65):12825-12829.
2. Rabai. K. A., O. H. Ahmed and S. Kasim, 2013. Use of formulated nitrogen, phosphorus, and potassium compound fertilizer using clinoptilolite zeolite in maize (*Zea mays*) cultivation. *Emirates Journal of Food and Agriculture*. **25** (9): 713-722.