UNIVERSITI PUTRA MALAYSIA

REDUCTION OF AMMONIA LOSS THROUGH MIXING UREA WITH ZEOLITE, PEAT SOIL WATER, AND SAGO WASTE WATER

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FSPM 2011 4

LATIFAH BTE OMAR MASTER OF SCIENCE 2011

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2011

DEDICATION

Dedicated to my family and friends who have supported and encouraged me throughout the preparation of this thesis

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirements for the degree of Master of Science

REDUCTION OF AMMONIA LOSS THROUGH MIXING UREA WITH ZEOLITE, PEAT SOIL WATER AND SAGO WASTE WATER

By

LATIFAH OMAR

May, 2011

Chairman : **Osumanu Haruna Ahmed, PhD**

Faculty : **Agriculture and Food Sciences**

Ammonia volatilization is one of the major mechanisms which lead to poor urea-N use efficiency. Two laboratory incubation experiments were conducted to study the effects of mixing urea with zeolite, peat soil water, and sago waste water on NH_3 volatilization, soil exchangeable NH_4^+, available NO_3^- contents compared with urea without additives under waterlogged and non-waterlogged conditions. The study compared seven different treatments which were: soil alone (T0), urea without additives (T1), urea mixed with 175 mL peat soil water and 0.75 g zeolite (T2), urea mixed with 175 mL peat soil water and 1.0 g zeolite (T3), urea mixed with 175 mL peat soil water (T4), urea mixed with 175 mL sago waste water and 0.75 g zeolite (T5), urea mixed with 175 mL sago waste water and 1.0 g zeolite (T6), and urea mixed with 175 mL sago waste water (T7). The mixtures (T2, T3, T4, T5, T6 and T7) significantly reduced NH_3

volatilization and also improved retention of soil exchangeable NH_4^+ compared with urea without additives (T1). All the mixtures of zeolite, sago waste water, and peat soil water with urea significantly increased soil exchangeable NH_4^+, available NO_3^- contents. The mixtures of sago waste water, zeolite, and peat soil water with urea delayed urea hydrolysis and NH_4^+ accumulation under waterlogged condition than under non-waterlogged condition. These mixtures were further tested (scale up) in a pot experiment (greenhouse) whereby maize (*Zea mays* L.) hybrid No. 5 was used as a test crop. The objectives of this greenhouse study were to evaluate the effects of the treatments on: (1) Nitrogen, P, and K uptake and their use efficiency in maize cultivation, and (2) Soil exchangeable NH_4^+, available NO_3^-, pH, exchangeable K, and available P contents. The treatments evaluated were: No fertilizer (T0), 2.02 g urea (T1), 2.02 g urea + 30 g zeolite + 7 L peat soil water (T2), 2.02 g urea + 40 g zeolite + 7 L peat soil water (T3), 2.02 g urea + 30 g zeolite + 7 L sago waste water (T4), 2.02 g urea + 40 g zeolite + 7 L sago waste water (T5), 2.02 g urea + 7 L peat soil water (T6) and 2.02 g urea + 7 L sago waste water (T7). Triple Super Phosphate (TSP) and Muriate of Potash (MOP) were used as sources of P and K for all the treatments except for T0. These fertilizers were applied twice that is 10 and 28 days after planting. Phosphorus and K requirements of the test crop were supplemented by applying TSP and MOP (standard rate for the test crop) to plants of all the treatments except T0. The mixtures of sago waste water, zeolite, and peat soil water with urea significantly improved maize dry matter, N, P, K uptake and N, P, K use efficiency compared with urea without additives. These mixtures also significantly increased soil exchangeable NH_4^+, K, Ca, Mg, available P, and NO_3^- contents compared with urea without additives. Amending urea with sago waste water and peat soil water can reduce NH_3 loss by encouraging formation of NH_4^+ and NO_3^- over NH_3. Additionally, the mixtures ensure N, P, K uptake and their use efficiency while at the same time making soil NH_4^+, K, Ca, Mg, P, and NO_3^- available for plant use.

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

PENGURANGAN PEMERUAPAN AMONNIA MELALUI CAMPURAN UREA DENGAN ZEOLITE, AIR TANAH GAMBUT DAN AIR SISA SAGU

Oleh

LATIFAH OMAR

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Pemeruapan ammonia ialah satu di antara mekanisme utama yang menyumbang kepada kelemahan keberkesanan penggunaan urea-N. Dua kajian makmal telah dijalankan bagi mempelajari kesan-kesan campuran urea dengan zeolite, air tanah gambut dan air sisa pemprosesan sagu ke atas pemeruapan NH_3, NH_4^+ tukarganti tanah, dan keterdapatan kandungan NO_3^- berbanding dengan urea tanpa sebarang campuran dalam keadaan tanah terendam dan bukan terendam. Kajian ini membandingkan tujuh rawatan berlainan yang terdiri daripada : tanah sahaja (T0), urea tanpa campuran (T1), urea dicampurkan dengan 175 mL air tanah gambut dan 0.75 g zeolite (T2), urea dicampurkan dengan 175 mL air tanah gambut dan 1.0 g zeolite (T3), urea dicampurkan dengan 175 mL air tanah gambut (T4), urea dicampurkan dengan 175 mL sisa air sagu dan 0.75 g zeolite (T5), urea

dicampurkan dengan 175 mL sisa air sagu dan 1.0 g zeolite (T6), dan urea dicampurkan dengan 175 mL sisa air sagu (T7). Campuran- campuran (T2, T3, T4, T5, T6 dan T7) dengan signifikan mengurangkan pemeruapan NH_3 dan juga meningkatkan penahanan NH_4^+ tukarganti tanah berbanding dengan urea tanpa campuran (T1). Semua campuran-campuran zeolite, air tanah gambut, dan air sisa sagu dengan urea secara signifikan meningkatkan kandungan NH_4^+ tukarganti tanah dan keterdapatan NO_3^-. Campuran-campuran ini juga mempunyai kesan besar dalam keupayaan untuk melambatkan hidrolisis urea dan pengumpulan NH_4^+ dalam keadaan terendam daripada keadaan bukan terendam. Campuran-campuran ini telah dikaji lebih lanjut (mengikut skala) dalam kajian pasu (rumah hijau) dimana jagung (*Zea mays* L.) hybrid No. 5 telah digunakan sebagai tanaman kajian. Matlamat kajian rumah hijau adalah untuk menilai kesan-kesan rawatan terhadap: (1) Pengambilan N, P dan K dan keberkesan penggunaan dalam penanaman jagung, dan (2) Ammonium tukarganti tanah, pH, kandungan-kandungan keterdapatan NO_3^-, tukarganti K, dan keterdapatan P. Rawatan-rawatan yang dinilai adalah: Tanpa baja (T0), 2.02 g urea (T1), 2.02 g urea + 30 g zeolite + 7 L air tanah gambut (T2), 2.02 g urea + 40 g zeolite + 7 L air tanah gambut (T3), 2.02 g urea + 30 g zeolite + 7 L air sisa sagu (T4), 2.02 g urea + 40 g zeolite + 7 L air sisa sagu (T5), 2.02 g urea + 7 L air tanah gambut (T6) and 2.02 g urea + 7 L air sisa sagu (T7). Triple Super Phosphate (TSP) dan Muriate of Potash (MOP) telah digunakan sebagai sumber-sumber P dan K bagi semua rawatan-rawatan kecuali bagi T0. Baja-baja ini telah digunakan dua kali iaitu pada 10 dan 28 hari selepas penanaman. Keperluan P dan K bagi tanaman kajian dipenuhi dengan menggunakan TSP dan MOP (kadar piawaian bagi tanaman kajian) pada semua tanaman bagi semua rawatan kecuali T0. Campuran-campuran air sisa sagu, zeolite, dan air tanah gambut dengan urea mempunyai kesan-kesan yang nyata ke atas bahan kering, pengambilan N, P, K, dan keberkesanan penggunaan N, P, K, berbanding dengan urea tanpa penambahan. Campuran-campuran ini juga nyata sekali mempertingkatkan NH_4^+ tukarganti tanah, kandungan-kandungan K, Ca, Mg, keterdapatan P dan NO_3^- berbanding dengan urea tanpa tambahan. Merawat urea dengan air sisa sagu dan air tanah gambut boleh mengurangkan kehilangan NH_3 dengan menggalakkan pembentukan NH_4^+ dan NO_3^- daripada NH_3. Tambahan lagi, campuran-campuran ini memastikan pengambilan N, P, K, dan keberkesanan penggunaannya dalam masa yang sama menjadikan NH_4^+, keterdapatan K, Ca, Mg, P, dan NO_3^- bagi kegunaan tanaman.

ACKNOWLEDGEMENTS

I would like to convey sincere appreciation to my supervisory committee, Associate Professor Dr. Osumanu Haruna Ahmed and Professor Dato' Dr. Nik Muhamad Ab. Majid for their encouragement, constant support and comments throughout this research.

I must thank my whole family especially my beloved mother Suyah Bt. Tigal, my brothers; Eduwine, Irsyadi Iman and Bero, my sisters; Sarina, Khadijah, Nurshazlin, Roslind, Mona and Eileen, not forgetting, Mr. Chong Ing Kiong (Mangkong), for their support and encouragement during this research.

I am grateful to Dr. Susilawati Kassim for her constructive suggestions. I am also thankful to Mr. Arni Japar and Mrs. Elizabeth Andrew for their laboratory assistance. Warmest thanks go to my friends, Ch'ng Huck Ywih, Auldry Chaddy, Taufik, Mawi Salimin, Regis Bernard, Norida Hanim, Latip Bundan, Mohd. Shukri, Suffian and Rakib Rashid for being helpful before, during and after my research. My appreciation also goes to the School of Graduate Studies and Universiti Putra Malaysia for financial support through the Graduate Research Fellowship.

I certify that a Thesis Examination Committee has met on 25 May 2011 to conduct the final examination of Latifah binti Omar on her thesis entitled "Reduction of Ammonia Loss through Mixing Urea with Zeolite, Peat Soil Water and Sago Waste Water" in accordance with the Universities and University College 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.

LATIFAH BT OMAR

Date: 25 May 2011

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LIST OF ABBREVIATIONS

N	Nitrogen
NH_3	Ammonia
$NH4^+$	Ammonium ions
NO_3^-	Nitrate
OH	Hydroxyl group
CEC	Cation Exchange Capacity
P	Phosphorus
K	Potassium
Ca	Calcium
Mg	Magnesium
H	Hydrogen
CO_3^{2-}	Carbonate ions
CO_2	Carbon dioxide
KCl	Potassium Chloride
NH_4OA_c	Ammonium acetate
HNO_3	Nitric acid
ppm	part per million
NaOH	Sodium hydroxide
SAS	Statistical analysis system
DNMRT	Duncan new multiple range test
HCl	Hydrochloric acid
COOH	Carboxyl group
$CaCO_3$	Calcium carbonate
Ha	Hectare
g	gram
mL	milliliter
MgO	Magnesium oxide
h	hour

°C	Celsius
NBPT	N-(n-butyl) thiophosphoric triamide
C	Carbon
mg	milligram
%	Percentage
ANOVA	Analysis of variance
$NaHCO_3$	Sodium bicarbonate
H_2SO_4	Sulphuric acid
K_2SO_4	Potassium sulphate
AAS	Atomic absorption spectrophotometer
NH_4Cl	Ammonium chloride
CsCl	Caesium chloride
OM	Organic matter
CRD	Complete randomized design
RCBD	Randomized complete block design
µm	micrometer
Kg	Kilogram
L	Liter
MOP	Muriate of potash
TSP	Triple superphosphate

CHAPTER 1

INTRODUCTION

Nitrogen (N) loss in soils occurs mainly due to ammonia (NH_3) volatilization, denitrification, leaching, and surface runoff (Brady and Weil, 2002). Ammonia volatilization is the process by which ammonia is lost from the earth's surface to the atmosphere. The exchange of NH_3 between soils, plants, water, and the atmosphere is an important part of the terrestrial nitrogen cycle (Saffigna and Freney, 2006). Ammonia is a readily identifiable product of nitrogen mineralization and formed continuously in soil and waterlogged areas.

Ammonia volatilization is an important pathway for fertilizer N loss from soil and also a major source of environmental pollution (Wang *et al*., 2004). Eutrophication of semi-natural ecosystems, formation of fine particular matter in the atmosphere, and alteration of the global greenhouse balance are some of the effects encountered when continous NH_3 gas enters the environment (Sutton *et al*., 2008). During hydrolysis, urea N is converted into NH_3 which subsequently reacts with a proton to produce ammonium ion (NH_4^+). Under alkaline condition, the equilibrium of $NH_3 + H_2O \rightleftharpoons NH_4^+ + OH^-$ shifts more to NH_3 increasing volatilization loss, a process which reduces N-use efficiency of urea (Sparks, 2005). Transport of gaseous NH_3 is recognized as important in the terrestial N cycle, and the formation of gaseous NH_3 in soil, plants and water can lead to considerable loss of N from agricultural land. As much as 70% of N fertilizers applied to the soil surface may be lost through NH_3 volatilization (Fenn and Kissel, 1975; Freney *et al*., 1983; Whitehead and Raistrick, 1990; Jayaweera and Mikkelsen, 1991). In addition, the gaseous NH_3 can be toxic to plants (Bremner, 1995).

The most used form of solid N fertilizer in recent times is urea. However, the reaction by which it forms is reversible: $(NH_2)_2CO + H_2O \rightleftharpoons 2\ NH_3 + CO_2$. The NH_3 produced from this reaction may be volatilize from surface-applied urea if it is not promptly incorporated into the the soil (Cai *et al*., 2002). The loss of urea N through volatilization reduces its effectiveness for surface application (Malhi *et al*., 1996). In a study to manage urea, Jones and Jacobsen (2005) found that in any other condition that moistens surface-applied urea without carrying it into the soil can cause it to hydrolyze.

Urea is the most competitive source of N fertilizer due to the easy handling, storage, transporation and production. It is a major source of inorganic N fertilizer as it account for 50% of the total world fertilizer N consumption (Sanz-Cobena *et al*., 2008). Worldwide used of urea is almost five times with that of NH_4NO_3 (Havlin *et al*., 2005). Ammonia volatilization lowers urea-N use efficiency in many cropping and pastures systems, meaning that a large percentage of the applied fertilizer N is not used for productive purposes and it is essentially lost (Bremner, 1995). After surface application, urea is readily hydrolyzed within 1 to 2 days by urease to NH_4^+, hydroxyl (OH^-) and carbonate (CO_3^{2-}) ions, leading to a high pH and very high concentrations of NH_4^+ around the urea granule. This NH_4^+ reaches equilibrium with the dissolved NH_3 near the soil surface. The sharp rise in soil pH (Zhengping *et al*., 1991) increases the likelihood of gaseous NH_3 loss to the atmosphere.

Broadcasting urea at the surface is a common practice in agriculture because of the ease of application and the low cost of urea. When applied at the soil surface, up to the 50% of the urea-N can be volatilized as NH_3 (Cai *et al*., 2002). Surface application of NH_4-N based fertilizer often results in considerable NH_3 volatilization (Sommer *et al*., 2004). Loss of fertilizer N through ammonia volatilization also occur from floodwater on a soil moderately to slightly acid, although losses are usually highest on alkaline soils (Sparks, 2005). Volatilization of NH_3 from a soil is a function of the various properties of the soil system involved, including moisture content, soil pH, cation exchange capacity (CEC), exchangeable cations, texture, lime material, temperature and atmospheric conditions above the soil (Freney *et al*., 1983; Fenn and Hossner 1985; De Datta *et al*., 1989). Agronomic variables such as rates and sources of N fertilizers and time, method and depth of application are also important (De Datta, 1981).

Subsequently, it is important to find appropriate methods of reducing problems encountered in the use of urea fertilizer, which include damage to seeds, seedlings and young plants, NO_3^- toxicity, phytotoxicity of foliar-applied urea, and volatilization of urea N as NH_3. Gaseous loss of urea fertilizer N as NH_3 is of particular concern because it can exceed 50% of the N applied (Gioacchini *et al*., 2002).

Several studies have been conducted to minimize the problems encountered in the use of urea fertilizer. Zeolites can be used to reduce NH_3 volatilization because of its high CEC and great affinity for NH_4^+ (Stumpe, 1984; Mackdown and Tucker, 1985; Ming and Dixon, 1986) thus the higher the CEC, the lower the NH_3 volatilization. Zeolites are important material which are widely used as sorbents, ion exchangers, catalysts and catalyst supports (Ming and Dixon, 1986). The unique feature of zeolites, which are framework structures with uniform pore systems, is their selective accessibility to sorbates and reactant molecules. They control the size of molecules adsorbed and the size of the reaction product (Ming and Dixon, 1986).

Some studies have also used acidic materials such as inorganic acids (phosphoric or nitric acid) for acidulation of urea (Gould *et al*., 1984; Fenn and Hossner, 1985; Ahmed *et al.,* 2009). The problem with this method is that the end product is corrosive and hence needs proper storage and handling. Watering soil with peat water and waste water from sago may help to reduce NH_3 loss from urea because of their acidic nature ranging from 3.2 to 4.0. The acidity of peat water is mainly due to organic acids such as humic and fulvic acid (Nurhidayah, 2007). The acidic nature of sago waste could also be exploited to reduce NH_3 loss from urea. Sago waste water has pH of 3.5, 38 g L^{-1} reducing and 20.2 g L^{-1} non-reducing sugars, and several minerals. Sago waste water is a waste product produced during processing of starch from sago trunks (Singh, 2006).

The following were hypothesized: i) Amending urea with zeolite, peat water and sago waste water will minimize ammonia volatilization as well as improving soil ammonium and nitrate, and ii) will improve urea-N use efficiency. There is dearth of information about the effectiveness of mixing urea with zeolite, peat soil water and water from sago waste on minimizing NH_3 volatilization from surface-applied urea. Thus this study was conducted to: (i) determine the effect of mixing urea with zeolite, peat soil water and waste water from sago on NH_3 loss and (ii) improve urea-N use efficiency by amending urea with zeolite, peat water and sago waste water.

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BIODATA OF STUDENT

Latifah Binti Omar was born on 2nd November 1972 at Dalat, Sarawak. She received her primary education from Sekolah Kebangsaan Sungai Ud Dalat, Sarawak. After completing her secondary school education at Sekolah Menengah Kerajaan Rosli Dhoby Sibu, Sarawak, she worked at Sekolah Menengah Kerajaan Three River Mukah Sarawak as a temporary teacher. She was registered as a full time diploma student at Universiti Putra Malaysia Bintulu Sarawak Campus and awarded a diploma in agriculture in 2005.

Latifah pursued her undergraduate programme at UPM Bintulu Sarawak Campus from 2005 to 2009 where she was awarded Bachelor Science in Bioindustry. In June 2009, she enrolled as a full time master's student at Universiti Putra Malaysia Bintulu Sarawak Campus. She had presented an oral paper at 1st Graduate Science Student Research Conference 2010 in Universiti Brunei Darussalam, Brunei Darussalam. Latifah has produced seven papers from her research work.

LIST OF PUBLICATIONS

1. Latifah, O., O.H. Ahmed, and A.M. Nik Muhamad. 2010. Minimizing ammonia volatilization in waterlogged soils through mixing urea with zeolite and sago waste water. International Journal of the Physical Sciences. Vol. 5(14): 2193-2197.

2. Latifah, O., O.H. Ahmed, and A.M. Nik Muhamad. 2011. Reducing ammonia loss from urea and improving soil exchangeable ammonium and nitrate in non-waterlogged soils through mixing zeolite and sago (*Metroxylon sagu*) waste water. International Journal of the Physical Sciences. Vol. 6(4): 866-870.

3. Latifah, O., O.H. Ahmed, and A.M. Nik Muhamad. 2011. Ammonia loss, ammonium and nitrate accumulation from mixing urea with zeolite and peat soil water under waterlogged condition. African of Journal Biotechnology. Vol. 10(7): 3365-3369.

4. Latifah, O., O.H. Ahmed, and A.M. Nik Muhamad. 2011. Ammonia loss, soil exchangeable ammonium and available nitrate contents from mixing urea with zeolite and peat soil water. International Journal of the Physical Sciences. Vol (6): 2916-2920.

5. Latifah, O., O.H. Ahmed, and A.M. Nik Muhamad. 2011. Effect of mixing urea with zeolite and sago (*Metroxylon sagu*) waste water on nutrient use efficiency of maize (*Zea mays* L.). The Scientific World of Journal. In review.

6. Latifah, O., O.H. Ahmed, and A.M. Nik Muhamad. 2011. Enhancing nutrient use efficiency of maize (*Zea mays* L.) from mixing urea with zeolite and peat soil water. International Journal of the Physical Sciences. In review.

7. Latifah, O., O.H. Ahmed, and A.M. Nik Muhamad. 2011. Effect of mixing urea with sago (*Metroxylon sagu*) waste water and peat soil water on minimizing ammonia volatilization and improvement of nutrient use efficiency of maize (*Zea mays* L.). The Scientific World of Journal. In review.

LIST OF AWARD

1. Bronze Medal, 'Green technology for efficient use of agricultural waste water'. An International Expo on Technology Invention and Innovation. Malaysian Association of Research Scientist, Malaysia Technology Expo 2011, Kuala Lumpur, Malaysia.

LIST OF PATENTS

<table>
<tr><th>No.</th><th>Owner</th><th>Innovator Name</th><th>Title</th><th>Status</th></tr>
<tr><td>1.</td><td rowspan="2">Universiti Putra Malaysia (UPM)</td><td rowspan="2">Osumanu Haruna Ahmed (UPM Bintulu Sarawak Campus)

Latifah Omar (UPM Bintulu Sarawak Campus)

Nik Muhamad Ab. Majid (UPM, Serdang Selangor, Malaysia)</td><td>An Improved Plant Treatment Agent.</td><td>Filed
Patent file number (PI2011002910)
Date of filing: 22/06/2011</td></tr>
<tr><td>2.</td><td>Improving Urea-N Efficiency Through the Use of Peat Water.</td><td>In progress</td></tr>
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