UNIVERSITI PUTRA MALAYSIA

PRODUCTION OF N, P, AND K HUMATES FROM PALM OIL MILL EFFLUENT SLUDGE AND THEIR EFFECTS ON CORN GROWTH

ROSLIZA SHAMSUDDIN

FSPM 2011 2

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By

ROSLIZA BINTI SHAMSUDDIN

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

October 2011

This Thesis is Dedicated to:
My Beloved Mother
Normah Bin Mohd Lazim
My Sisters and Brother
Shamsina, Norhaslinda and Azlan
And the one who cares
Thank you for your love and support

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

PRODUCTION OF N, P, AND K HUMATES FROM PALM OIL MILL EFFLUENT SLUDGE AND THEIR EFFECTS ON CORN GROWTH

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October 2011

Chairman : Associate Professor Ahmed Osumanu Haruna, PhD

Faculty : Agriculture and Food Sciences (Bintulu)

Substantial loss through ammonia volatilization from surface-applied urea reduces urea-N use efficiency in plant production, pollutes environment and burdens economy. This problem could be minimized through the use of agricultural wastes such as palm oil mill effluent (POME) sludge. POME sludge could reduce this volatilization in view of its acidic nature and high cation exchange capacity (CEC) present in it the form of humic acids (HA) and fulvic acids (FA). The objectives of this study were to: (I) Evaluate if N, P and K humates produced from POME sludge could minimize ammonia volatilization from urea as well as improving retention of soil exchangeable ammonium and nitrates in laboratory condition and (ii) Evaluate the effect of N, P, and K humates on soil pH, exchangeable ammonium, available nitrate; N, P, and K concentrations, their uptake and use efficiency in corn (*Zea mays*) cultivation under greenhouse condition.

HA and FA were isolated from air-dried POME sludge using 0.5 M KOH and 6 N H_2SO_4. Daily ammonia loss was measured for 15 days using a closed-dynamic air flow system. Ammonia released was estimated using boric acid indicator traps which were replaced every 24 h and titrated with 0.01 N HCl. Soil samples were evaluated for pH, exchangeable ammonium and available nitrate. Under greenhouse condition, formulated fertilizers were applied 10 days after planting (DAP) in each pot containing 10 kg of soil. Soil and plant samples (stems, leaves and roots) were sampled at 31 DAP. Soil samples were analyzed for pH, ammonium and nitrate content. Plant samples were analyzed for dry matter production, N, P, and K concentrations, N, P, and K uptake, and their use efficiency. From the laboratory study, Urea-TSP-MOP-HA, Urea-TSP-MOP, urea-FA, Urea-TSP-MOP-FA, Urea-acidified (HA+FA) and Urea-TSP-MOP-acidified (HA+FA) mixtures significantly reduced ammonia by 13, 20, 23, 30%, and 100% (of the N added as urea), respectively. These treatments also caused significant retention of soil exchangeable ammonium. When the treatments were further tested in a greenhouse, Urea-TSP-MOP, urea-FA, Urea-TSP-MOP-Humin, Urea-TSP-MOP-acidified(HA+FA), Urea-TSP-MOP-FA and Urea-TSP-MOP-HA mixtures had significant effect on dry matter production, N, P, and K uptake and use efficiency of the test crop (*Zea mays*) compared to urea alone. Urea amended with HA, FA, TSP and MOP minimized ammonia volatilization, as well as improving soil exchangeable ammonium, and urea-N use efficiency. It must be stressed that results obtained in the laboratory and greenhouse experiments may only be applicable to similar acid soils.

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

**PENGHASILAN N, P, DAN K HUMAT
DARIPADA MENDAKAN EFFLUEN KILANG MINYAK SAWIT
DAN KESANNYA PADA PERTUMBUHAN JAGUNG**

Oleh

ROSLIZA BINTI SHAMSUDDIN

Oktober 2011

Pengerusi : Profesor Madya Ahmed Osumanu Haruna, PhD

Fakulti : Fakulti Sains Pertanian dan Makanan (Bintulu)

Kerugian besar melalui pemeruapan amonia dari urea yang ditabur atas tanah mengurangkan kecekapan penggunaan N-urea bagi pengeluaran tanaman, mencemarkan alam sekitar, dan membebankan ekonomi. Masalah ini dapat diminimumkan melalui penggunaan sisa pertanian seperti mendakan effluen kilang minyak sawit (POME). Mendakan POME dapat mengurangkan pemeruapan ini melihat kepada sifat keasidan semula jadinya dan kadar pertukaran kation (KPK) yang tinggi hadir di dalamnya dalam bentuk asid humik (HA) dan asid fulvik (FA). Antara objektif kajian adalah : (i) Menilai jika N, P dan K Humat yang dihasilkan dari mendakan POME dapat mengurangkan pemeruapan amonia dari urea selain mempertingkatkan penahanan amonium dan nitrat dalam tanah dalam keadaan makmal dan (ii) Menilai kesan N, P dan K humat pada pH, amonium, nitrat tanah; kepekatan N, P dan K, pengambilan N, P dan K, dan kecekapan penggunaannya oleh tanaman jagung (*Zea mays*) dalam keadaan rumah hijau.

HA dan FA dipencilkan daripada mendakan POME kering menggunakan 0.5 KOH dan 6 N H_2SO_4. Kehilangan amonia secara hariannya disukat selama 15 hari menggunakan satu kaedah sistem pengaliran udara dinamik pada keadaan tertutup. Amonia yang terbebas disukat menggunakan perangkap penunjuk asid borik yang mana diganti setiap 24 jam dan dititrat menggunakan 0.01 N HCl. Sampel tanah dinilai untuk pH, amonium dan nitrat. Dalam keadaan rumah hijau, baja yg diformulasi diaplikasikan 10 hari selepas ditanam (HST) pada setiap pasu yang mengandungi 10 kg tanah. Sampel tanah dan tanaman (batang, daun, dan akar) disampel pada 31 HST. Sampel tanah dianalisis untuk pH, kandungan amonium dan nitrat. Sampel tanaman dianalisis untuk pengeluaran jirim kering, kepekatan N, P, dan K, pengambilan N, P dan K, dan kecekapan penggunaannya. Hasil kajian makmal menunjukkan campuran Urea-TSP-MOP-HA, Urea-TSP-MOP, urea-FA, Urea-TSP-MOP-FA, Urea-asidik (HA+FA) dan Urea-TSP-MOP-asidik (HA+FA) mengurangkan amonia secara signifikan masing-masing dengan 13, 20, 23, 30%, dan 100% (dari N yang ditambah dari urea). Rawatan ini juga mengakibatkan penahanan amonium yang signifikan dalam tanah. Apabila rawatan dikaji selanjutnya dalam keadaan rumah hijau, campuran Urea-TSP-MOP, urea-FA, Urea-TSP-MOP-Humin, Urea-TSP-MOP-asidik (HA+FA), Urea-TSP-MOP-FA dan Urea-TSP-MOP-HA menunjukkan kesan signifikan terhadap pengeluaran jirim kering, pengambilan N, P, dan K dan kecekapan kegunaan oleh tanaman ujian (*Zea mays*) berbanding urea sahaja. Urea dicampur dengan HA, FA, TSP dan MOP mengurangkan pemeruapan amonia, selain mempertingkat amonium tanah dan kecekapan penggunaan N-urea. Ia harus ditekankan bahawa keputusan yang diperolehi dalam eksperimen makmal dan rumah hijau hanya boleh diaplikasi pada tanah berasid yang serupa sahaja.

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I certify that a Thesis Examination Committee has met on 7 October 2011 to conduct the final examination of Rosliza binti Shamsuddin on her thesis entitled "Production of N, P, and K Humates from Palm Oil Mill Effluent Sludge and their effects on corn growth" in accordance with the Universities and Universities 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 Agronomy.

Members of the Thesis Examination Committee were as follows:

Japar Sidik Bujang, PhD
Professor
Faculty of Agriculture and Food Science
Universiti Putra Malaysia Bintulu Campus
(Chairman)

Shamsuddin Jusop, PhD
Professor
Faculty of Agriculture
Universiti Putra Malaysia
(Internal Examiner)

Siva K Balasundram, PhD
Lecturer
Faculty of Agriculture
Universiti Putra Malaysia
(Internal Examiner)

Kopli Bujang, PhD
Professor
Centre of Graduate Studies
Universiti Malaysia Sarawak
(External Examiner)

BUJANG BIN KIM HUAT, PhD
Professor and Deputy Dean
School of Graduate Studies
Universiti Putra Malaysia

Date: 20 January 2012

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

Ahmed Osumanu Haruna, PhD
Associate Professor
Faculty of Agriculture and Food Sciences
Universiti Putra Malaysia Bintulu Campus
(Chairman)

Nik Muhamad Abd Majid, PhD
Professor Dato
Faculty of Forestry
Universiti Putra Malaysia
(Member)

BUJANG BIN KIM HUAT, PhD
Professor and Dean
School of Graduate Studies
Universiti Putra Malaysia

Date:

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 other institution.

ROSLIZA BINTI SHAMSUDDIN

Date: 7 October 2011

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LIST OF ABBREVIATIONS TERMS

1 **ANOVA** – Analysis of Variance

2 **AAS** – Atomic Absorption Spectrophotometry

3 **BOD** – Biological Oxygen Demand

4 **BD** – Bulk Density

5 **C** – Carbon

6 **Ca** – Calcium

7 **CEC** – Cation Exchange Capacity

8 **CO_2** – Carbon Dioxide

9 **COD** – Chemical Oxygen Demand

10 **DAP** – Days After Planting

11 **FA** – Fulvic Acids

12 **G -** Gravity

13 **H** – Hydrogen

14 **HS** – Humic Substances

15 **h** – hour

16 **H_2SO_4** – Sulphuric Acid

17 **H_3BO_3** – Boric Acid

18 **HA** – Humic Acids

19 **HCl** – Hydrochloric Acid

20 **HS** – Humic Substances

21 **K** – Potassium

22 **KBr** – Potassium Bromide

KOH – Potassium Hydroxide

KCl – Potassium Chloride

M - Molarity

Mg – Magnesium

MgO – Magnesium oxide

min - minute

MOP – Muriate of Potash

N – Nitrogen

Na – Sodium

NaCl – Sodium Chloride

$NaHCO_3$ – Sodium Bicarbonate

NaOH – Sodium Hydroxide

NH_3 – Ammonia

NH_4 – Ammonium

NH_4OAc – Ammonium acetate

NO_3 – Nitrate

NO_2 – Nitrite

NUE - Nutrient Use Efficiency

O – Oxygen

OM – Organic Matter

P – Phosphorus

POME – Palm Oil Mill Effluent

ppm – part per million

RCBD – Randomized Complete Block Design

47 **rpm** – Rotation per minute

48 **S** – Sulphur

49 **SAS** – Statistical Analysis System

50 **sec** - second

51 **TOC** – Total Organic Carbon

52 **TSP** – Triple Superphosphate

CHAPTER 1

INTRODUCTION

Malaysian palm oil production may reach 18.8 million tons in 2011 where global palm oil production may rise by 5.5 million tons in 2011 and 2 million tons in 2012 (Bloomberg, 2011). In 2008, palm oil production was 17.7 million tons on 4.5 million hectares (Malaysian Palm Oil Industry Performance, 2008). Malaysian palm oil exports increased to 16.7 million tons in 2010, worth 59.8 billion ringgits, compared with the 15.9 million tons that were shipped in 2009, according to data from the Malaysian Palm Oil Board (Bloomberg, 2011). The export earnings of more than RM 68 billion in 2007 have been reported by the World Bank and the Asian Development Bank (Bernama, 2009). With such large production of palm oil there would also be abundant by-products such as palm oil mill effluent (POME).

POME contains high bio-chemical oxygen demand BOD (25,000 mg/L) and chemical oxygen demand COD (50,000 mg L^{-1}) content that pose a great threat to water environment (Yeow, 1983), making it about 100 times more polluting than domestic sewage (Okwute and Isu, 2007). In terms of its population equivalent, the BOD generated by the palm oil industry in Malaysia in 1998 is equivalent to that generated by 38 million people (Bek-Nielsen *et al.,* 1999). This contributes 83% of the single largest polluter of the palm oil industry in Malaysia (Kwon *et al.,* 1989). Disposal of this highly polluting waste is an economic burden on communities and industries (O-Thong *et al.,* 2008), therefore, adding value to this waste could be economically viable.

POME could be put into good use in view of its high content of organic matter (Ahmad *et al.,* 2003) potentially present in the form of humic acids (HA) and fulvic acids (FA). These acids have a great potential to control ammonia volatilization since they are acidic materials which aid to lower drastic increase in pH at the soil microsite when urea is surface-applied. Ammonia volatilization from urea is known to be the major reason for economic loss of N for years (Bock and Kissel, 1998; Cai *et al.,* 2002; Brady and Weil, 2002). Ammonia loss is serious when it is surface-applied to a sandy soil as this soil has low cation exchange capacity (CEC) that decrease the soil's ability to hold exchangeable NH_4^+, thereby increasing the total amount of ammoniacal N in solution which is subject to volatilization (Fan and Mackenzie, 1993).

High total acidity (is also used as a measure for the CEC (Tan, 2003)) associated with HA may aid to retain NH_4^+ and NO_3^- (Stumpe *et al.,* 1984; Siva *et al.,* 1999; Ahmed *et al.,* 2006a; Ahmed *et al.,* 2006b) which are the plant usable form of N. The exchange capacity of FA is twice that of HA due to the total number of carboxyl (COOH) groups present and this could help retain more NH_4^+ and NO_3^-. High contents of NH_4^+ and NO_3^- without good retention may not guarantee plant N use efficiency because both NH_4^+ and NO_3^- are prone to leaching (Brady and Weil, 2002). Additional loss of N from soil is caused by the biological transformation of NH_4^+ and NO_3^- under anaerobic condition and denitrification process of converting NO_3^- to N_2 (Fan and Mackenzie, 1993). Good retention of ammonium ions (the common source of both nitrification-denitrification and volatilization) could enhance the efficiency of urea.

In some studies, acidic materials such as HA and TSP have been used to reduce ammonia loss from surface-applied urea (Stumpe *et al.*, 1984; Siva *et al.*, 1999; Ahmed *et al.*, 2006a; Ahmed *et al.*, 2006b) but an information such as this is lacking for POME sludge. These acidic materials lower the soil micro-site pH immediately around the fertilizer, a process which reduces hydrolysis of urea thus reducing ammonia loss. The use of muriate of potash (MOP) may help prevent the deficiency of chlorine under field conditions since chlorine is only very weakly adsorbed to soil colloids (Fan and Mackenzie, 1993). The mixtures containing MOP, triple superphosphate (TSP) and urea could also reduce ammonia loss, retain exchangeable NH_4^+ and NO_3^- in soil and also serve as a slow release complete fertilizer. This study may improve urea N use efficiency as well as reducing environmental pollution.

Objectives

The objectives of this study were:

(1) To evaluate if N, P, and K humates produced from POME sludge could minimize ammonia volatilization from urea as well as improving retention of soil exchangeable ammonium and available nitrate; and

(2) To evaluate the effect of N, P, and K humates on soil pH, exchangeable ammonium, available nitrate; N, P and K concentrations, their uptake and use efficiency in corn (*Zea mays*) cultivation under greenhouse condition.

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BIODATA OF STUDENT

Rosliza binti Shamsuddin was born in Kuantan, Pahang. She received her primary education at the Sekolah Kebangsaan Permatang Badak, Kuantan and her secondary education at Sekolah Menengah Abdul Rahman Talib (SMART), Kuantan and Maktab Rendah Sains Mara (MRSM), Kuantan. At school, she was actively involved in athletics, games, and other co-curricular activities. She was Pahang bowler for 5 years. After passing her Malaysia Certificate of Education in 2002, she went to Perak Matriculation College, Gopeng Perak (KMPK) and had a band 4 in Malaysian University English Test (MUET) in 2003.

She pursued her undergraduate study at Universiti Putra Malaysia Bintulu Campus, Sarawak (UPMKB) in 2004. She was very active in UPMKB Taekwando WTF Club and had a black belt. She went for two months practical training at Food Technology Centre, MARDI, Serdang, Selangor, Malaysia. In 2008, she was honoured with a second class upper for her Bachelor Degree in Bioindustry. From her final year project, she had published a journal article entitled "Simple and Rapid Method of Isolating Humic Acids from Tropical Peat Soils (Saprists)" in American Journal of Applied Sciences.

She worked as a Research Assistant (RA) from June-July 2008 on a project entitled "Development of organic based K foliar fertilizer from peat soils of Malaysia".

She pursued her Master of Science in Agronomy programme in July 2009 and was funded by the Graduate Research Fellowship (GRF). As a GRF recipient, she was required to assist in teaching, tutoring, supervising final year students or laboratory classes for duration of 4 hours per week including during semester break. Prior to the first semester, the courses that she assisted included Wastes Management and Utilization (KKA 4101), Experimental Design and Analysis (BIP 3402), Advanced Biostatistic (BGY 5404), and General Chemistry (CHM 2000) at both undergraduate and graduate levels.

She worked on a thesis entitled "The Production of N, P, and K Humates from Palm Oil Mill Effluent POME Sludge and their Effects on Corn Growth". Her roles as a researcher has proven to be very productive based on achievements made by her and her research team. She has filed a patent based research conducted during her candidature entitled. She has won 4 awards both locally and internationally. She has published 2 journal articles and a proceeding. She participated in two exhibitions. She performed exceptionally well as a postgraduate student.

LIST OF PUBLICATIONS

Publications

1) **Rosliza, S.**, O.H. Ahmed and A.M. Nik Muhamad. 2009. Reduction of Ammonia Volatilization through Mixing Urea with Humic and Fulvic Acids isolated from Palm Oil Mill Effluent Sludge. *American Journal of Environmental Sciences* 5(3): 382-386, 2009. ISSN 1553-345X.

2) **Rosliza, S.**, O.H. Ahmed and A.M. Nik Muhamad. 2009. Controlling Ammonia Volatilization and improving soil-exchangeable ammonium and nitrate retention by Mixing Urea with Humic Acid, Fulvic Acid, Tripple superphosphate and Muriate of Potash. *American Journal of Environmental Sciences* 5(5): 605-609, 2009. ISSN 1553-345X.

Awards

3) **Gold Medal**: Ahmed, O.H., **Rosliza, S.**, Susilawati K., Nik Muhamad A.M., Y. Khanif and H. Aminudin. 2009. Simple Techniques for Zero Ammonia Pollution from Ammonia Based Fertilizers. Pameran Reka Cipta, Penyelidikan dan Inovasi UPM 2009, Pusat Kebudayaan dan Kesenian Sultan Salahuddin Abdul Aziz Shah UPM Serdang, Malaysia.

4) **Excellent Poster Presentation Award**: **Rosliza, S.**, O.H. Ahmed and A.M. Nik Muhamad. 2009. Reduction of Ammonia Volatilization by Mixing with Humic and Fulvic Acid. Soil Science Conference Malaysia 2009, Kuala Terengganu, Malaysia.

5) **Bronze Medal: PECIPTA, 8-10 October 2009, KLCC, Malaysia.** Ahmed, O.H., **Rosliza, S.**, Auldry, C.P., Mohd Taufik M.Y., Susilawati K., A.M. Nik Muhamad and H. Aminuddin. 2009. Sustainable Technology for Reduction of Ammonia Pollution in the Environment.

6) **Gold Medal: IENA, 5-8 November 2009, Nuremberg, Germany.** Ahmed, O.H., **Rosliza, S.**, Mohd Taufik M.Y., Susilawati K., A.M. Nik Muhamad and H. Aminuddin. 2009. Simple, rapid and cost effective technology for producing organic based fertilizers.

Patent

7) Pending patent: Ahmed, O.H., **Rosliza, S.**, Susilawati K., A.M. Nik Muhamad and H. Aminuddin. "Reduction of ammonia loss from urea using humic and fulvic acids extracted from palm oil mill effluent sludge", ICC, UPM. Date of approval at ICC, UPM, 6th October 2009.

Exhibitions

8) **IENA, 5-8 November 2009, Nuremberg, Germany.** Ahmed, O.H., **Rosliza, S.**, Mohd Taufik M.Y., Susilawati K., A.M. Nik Muhamad and H. Aminuddin. 2009. Simple, rapid and cost effective technology for producing organic based fertilizers.

9) **PECIPTA, 8-10 October 2009, KLCC, Malaysia.** Ahmed, O.H., **Rosliza, S.**, Auldry, C.P., Mohd Taufik M.Y., Susilawati K., A.M. Nik Muhamad and H. Aminuddin. 2009. Sustainable Technology for Reduction of Ammonia Pollution in the Environment.

Proceeding

10) **Rosliza, S.**, O.H. Ahmed and A.M. Nik Muhamad. 2009. Reduction of Ammonia Volatilization by Mixing with Humic and Fulvic Acid. Soil Science Conference Malaysia 2009, Kuala Terengganu, Malaysia.

Manuscript for publication in cited international journal

11) **Rosliza, S.**, O.H. Ahmed and A.M. Nik Muhamad. 2009. Effect of N, P and K Humates on Ammonia Volatilization Reduction and Soil Exchangeable Ammonium and Nitrate Retention under Laboratory Condition. *American Journal of Applied Sciences*.

12) **Rosliza, S.**, O.H. Ahmed and A.M. Nik Muhamad. 2010. Effect of N, P, and K Humates on Dry Matter of *Zea mays* and Soil Exchangeable Ammonium and Available Nitrate. *American Journal of Applied Sciences*.

13) **Rosliza, S.**, O.H. Ahmed and A.M. Nik Muhamad. 2011. Effect of N, P, and K Humates on Selected Soil Chemical Properties, N, P, and K Contents in *Zea mays* Tissues, their Uptake, and Use Efficiency. *American Journal of Applied Sciences.*