

UNIVERSITI PUTRA MALAYSIA

USE OF CLINOPTILOLITE ZEOLITE TO IMPROVE EFFICIENCY OF PHOSPHORUS USE IN ACID SOILS

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By

NUR AAINAA BINTI HASBULLAH

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

June 2016

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June 2016

Chairman : Ahmed Osumanu Haruna, PhD Faculty : Agriculture and Food Sciences (Bintulu)

In acid soil of the tropics, soluble P is fixed by aluminium (AI) and iron (Fe). Therefore, efficient management of P fertilizers is critical to meet crops demand and to as well ensure good yield of crops and adequate food supply. In addition, mitigating environmental impacts on water quality and conservation of finite P deposit cannot be over emphasized. Clinoptilolite zeolite as an amendment could be used to mitigate P fixation in acid soils. To this end, a series of experiments were conducted including incubation study, pot trial, and two cycles of Zea mays L. cultivation at Universiti Putra Malaysia, Bintulu Sarawak Campus, Malaysia to improve P use efficiency and to also reduce amount of fertilizers (N, P, and K) use by amending fertilizers with Clinoptilolite zeolite. The three P fertilizers used in this study were a highly soluble P fertilizer (Triple superphosphate, TSP), rock phosphate fertilizers (Christmas Island rock phosphate, CIRP), and Egypt rock phosphate (ERP). The test crop used in this study was F1 maize hybrid (Hibrimas). In the incubation study, different amounts of Clinoptilolite zeolite and fertilizers were evaluated in a controlled environment. The treatments evaluated were: soil alone (T0), 100% fertilizer recommended rates (T1, E1, and C1), 75% fertilizer + 85% Clinoptilolite zeolite based on weight of fertilizer (T2, E2, and C2), 50% fertilizer + 100% Clinoptilolite zeolite (T3, E3, and C3), and 25% fertilizer rate + 115% Clinoptilolite zeolite (T4, E4, and C4). Soil pH was significantly improved with Clinoptilolite zeolite inclusion, whereas soil exchangeable Ca, Mg, Al, and soil acidity were comparable to the recommended rates. Decreased trend in soil exchangeable K, total P, and available P is related to the fertilizers reduction. Generally, P availability and reduction of P fixation (Al-P, Fe-P, Ca-P, reductant-P, and occluded-P) were not significant (inconsistent) in this incubation study, the effect could be different with plant interaction. These aspects were tested in a pot study. Treatments with 25% fertilizer reduction (T2, E2, and C2) were chosen as they showed the closest effects on selected

soil chemical properties as compared to the recommended fertilizer rates. The pot study conducted in a controlled environment revealed that amending reduced amounts of fertilizers with Clinoptilolite zeolite had similar effects on selected soil chemical properties and plant performance (dry matter production, nutrients uptake, and nutrients use efficiency). Amount of P fixed was similar to the recommended fertilizer rates thus, explaining the lack of differences in soil P availability, total P, plant dry matter production, nutrients uptake, and nutrients use efficiency despite 25% fertilizer reduction. This suggests the beneficial effect of Clinoptilolite zeolite in reducing P fixation besides improving nutrient uptake and use efficiency. The potential of Clinoptilolite zeolite and its effects on soil chemical properties and Zea mays L. productivity were further determined in a field trial. In the two field trials, maize plants dry matter production, nutrients uptake, and agronomic efficiency were similar regardless of fertilizer rate. Yield of fresh cobs in the first plant cycle showed that the recommended rates of TSP (T1), ERP (E1), and CIRP (C1) were 17 t ha⁻¹, 9.1 t ha⁻¹, and 8.8 t ha⁻¹, respectively. Reducing fertilizers by 25% but with Clinoptilolite zeolite resulted in comparable fresh cob yield as that of the recommended fertilizer rates. In the second plant cycle, there was an increase in the fresh cob yield. Plots with TSP and the treatments with fertilizers reduction and Clinoptilolite zeolite yielded 25 t ha⁻¹ and 22 t ha⁻¹ fresh cobs, respectively. Application of ERP resulted in 11.6 t ha⁻¹ in both recommended and reduction treatments whereas CIRP recorded 17.5 t ha⁻¹ for the recommended treatment and 15 t ha⁻¹ fresh cobs for the reduced fertilizer rate. Clinoptilolite zeolite inclusion in the first plant cycle neither increased soil pH, P availability, and basic cations nor reduced P fixation, soil acidity, and exchangeable AI. The aforementioned results remained similar in the second cycle of maize cultivation except for pH. Inclusion of Clinoptilolite zeolite increased soil pH as the recommended fertilizer rates. Although Clinoptilolite zeolite inclusion neither improved P availability nor reduced P adsorption, similar retention and availability of P despite 25% fertilizer reduction was observed. This suggests that Clinoptilolite zeolite enhanced-exchange mechanism, retention of basic cations from leaching, RP dissolution, efficient use of fertilizer thus, producing desirable yield. Clinoptilolite zeolite is beneficial and could be used to reduce the amount of N, P, and K fertilizers use in Zea mays L. cultivation on acid soils besides reducing the risk of environmental pollution. Perhaps, in a long term application, selected soil chemical properties could be significantly improved through the conditioning effects of Clinoptilolite zeolite. Clinoptilolite zeolite application reduced leaching losses of Ca and Mg, hence, the similar results obtained in this study regardless of treatment. Availability of N, P, K, and Fe in soil significantly reduced with Clinoptilolite zeolite application with 25% fertilizer reduction. As proven in the two cycles of maize cultivation, the use of Clinoptilolite zeolite in agriculture is beneficial as it can be used to reduce the unbalanced use of N, P, and K fertilizers of Zea mays L. and related crops cultivated on acid soils. Besides, it can be used to minimize environmental pollution due to excessive use of chemical fertilizers and mobility of toxic elements. Economic viability analysis for including Clinoptilolite zeolite in maize cultivation revealed that the total cost of production reduced with Clinoptilolite zeolite adoption due to elimination of liming. Benefit cost-ratio for the recommended fertilizer rates was 1.69 whereas Clinoptilolite zeolite inclusion increased benefit cost-ratio (1.83-1.84). Thus, adoption of Clinoptilolite zeolite in reduced amount of fertilization was found to be economically feasible as it not only gives higher profit in return but hopefully could also promote sustainability of agricultural productivity and soil fertility.



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

PENGGUNAAN ZEOLIT KLINOPTILOLIT MENINGKATKAN KECEKAPAN PENGGUNAAN FOSFORUS DI TANAH BERASID

Oleh

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Di dalam tanah berasid di kawasan tropika, fosforus (P) larut diikat oleh aluminium (Al) dan ferum (Fe). Oleh itu, pengurusan yang cekap bagi baja P adalah kritikal untuk memenuhi permintaan tanaman dan juga memastikan hasil tanaman yang baik dan bekalan makanan yang mencukupi. Tambahan pula, pengurangan kesan alam sekitar seperti kualiti air dan pemuliharaan deposit P vang terhad tidak boleh diabaikan. Zeolit Klinoptilolit sebagai bahan penambahbaik boleh digunakan untuk mengurangkan pengikatan P dalam tanah berasid. Untuk tujuan ini, beberapa siri eksperimen termasuk kajian inkubasi, kajian pasu, dan dua pusingan penanaman jagung hibrid F1 (Zea mays L.) telah dijalankan di Universiti Putra Malaysia, Kampus Bintulu Sarawak, Malaysia untuk meningkatkan kecekapan penggunaan P dan juga mengurangkan penggunaan baja (N, P, dan K) dengan menambahbaik baja P dengan Zeolit Klinoptilolit. Tiga baja P yang digunakan dalam kajian ini adalah baja P tersangat larut (Triple superphosphate phosphate, TSP), baja batuan fosfat (batuan fosfat Pulau Krismas, CIRP), dan batu fosfat Mesir (ERP). Tanaman yang digunakan dalam kajian ini adalah jagung hibrid F1 (Hibrimas). Dalam kajian inkubasi, jumlah Zeolit Klinoptilolit dan baja yang berbeza telah diuiji di dalam persekitaran yang terkawal. Rawatan yang dinilai jalah: tanah sahaja (T0), 100% kadar baja yang disyorkan (T1, E1, dan C1), 75% baja + 85% Zeolit Klinoptilolit (T2, E2, dan C2), 50% baja + 100% Zeolit Klinoptilolit (T3, E3 dan C3), dan 25% kadar baja + 115% Zeolit Klinoptilolit (T4, E4, dan C4). pH tanah telah meningkat secara bererti dengan kehadiran Zeolit Klinoptilolit, manakala Ca tukar ganti, Mg, Al, dan keasidan tanah adalah sama dengan kadar baja yang disyorkan. Trend menurun untuk K tukar ganti, total P, dan P tersedia dalam tanah adalah ekoran dari pengurangan baja. Umumnya, ketersediaan P dan pengurangan pengikatan P (AI-P, Fe-P, Ca-P, P-reductant, dan P-terperangkap) adalah tidak ketara dalam kajian inkubasi ini. Aspek-aspek ini kemudiannya telah diuji dalam kajian pasu. Rawatan dengan 25% pengurangan baja (T2, E2, dan C2) telah dipilih kerana ianya telah memberikan kesan yang hampir sama terhadap sifat kimia

tanah jika dibandingkan dengan kadar baja yang disyorkan. Kajian pasu yang telah dijalankan dalam persekitaran terkawal mendapati bahawa menambahbaik jumlah baja yang dikurangkan dengan Zeolit Klinoptilolit mempunyai kesan yang sama ke atas sifat kimia tanah terpilih dan prestasi tanaman (penghasilan berat kering, pengambilan nutrien, dan kecekapan penggunaan nutrien). Jumlah pengikatan P adalah sama dengan kadar baja yang disyorkan. Oleh itu ianya menjelaskan tentang perbezaan untuk P tersedia, total P, pengeluaran berat kering, pengambilan nutrien, dan kecekapan penggunaan nutrien meskipun 25% baja telah dikurangkan. Ini menunjukkan bahawa Zeolit Klinoptilolit adalah berfaedah dalam mengurangkan pengikatan P disamping meningkatkan pengambilan nutrien, dan kecekapan penggunaannya. Potensi Zeolit Klinoptilolit dan kesannya terhadap sifat kimia tanah dan produktiviti tanaman jagung (Zea mays L.) seterusnya diuji dalam kajian lapangan. Di dalam dua kajian lapangan, pengeluaran berat kering jagung, pengambilan nutrien, dan kecekapan agronomi adalah sama tanpa mengira kadar baja. Hasil tongkol segar jagung dalam pusingan pertama penanaman menunjukkan bahawa kadar TSP (T1), ERP (E1), dan CIRP (C1) yang disyorkan masing-masing memberikan hasil 17 t ha⁻¹, 9.1 t ha⁻¹, dan 8.8 t ha⁻¹. Pengurangan baja sebanyak 25% tetapi penambahan Zeolit Klinoptilolit telah menghasilkan tongkol segar yang setanding dengan kadar baja yang disyorkan. Di dalam pusingan kedua, terdapat peningkatan dalam hasil tongkol segar jagung. Plot dengan rawatan TSP pada kadar yang disyorkan dan rawatan baja kadar baja pada kadar yang dikurangkan dengan penambahan Zeolit Klinoptilolit masing-masing memberikan hasil sebanyak 25 t ha⁻¹ dan 22 t ha¹ tongkol segar. Penggunaan ERP menghasilkan 11.6 t ha¹ di dalam keduadua kadar yang disyorkan dan rawatan dengan pengurangan baja CIRP merekodkan sebanyak 17.5 t ha⁻¹ untuk baja yang disyorkan dan 15 t ha⁻¹ tongkol segar untuk kadar baja yang dikurangkan. Penambahan Zeolit Klinoptilolit dalam pusingan pertama tidak meningkatkan pH tanah, P tersedia, kation asas, dan tidak juga mengurangkan penjerapan P, keasidan tanah, dan Al tukar ganti. Keputusan yang disebutkan di atas kekal sama dalam pusingan kedua penanaman jagung kecuali pH. Penambahan Zeolit Klinoptilolit telah meningkatkan pH tanah setara dengan kadar baja yang disyorkan. Meskipun penambahan Zeolit Klinoptilolit tidak meningkatkan ketersediaan P mahupun mengurangkan penjerapan P, 25% baja telah dikurangkan dan P yang dipegang dan ketersediaan P adalah sama. Ini menunjukkan bahawa Zeolit Klinoptilolit telah meningkatkan mekanisme pertukaran, pemegangan kation asas dari dilarutlesap, kelarutan RP, dan kecekapan penggunaan baja dengan memberikan hasil tanaman yang sama . Zeolit Klinoptilolit adalah bermanfaat dan boleh digunakan untuk mengurangkan penggunaan baja N, P, dan K di dalam penanaman jagung (Zea mays L.) di tanah berasid disamping mengurangkan risiko pencemaran alam sekitar. Berkemungkinan, aplikasi jangka masa panjang akan mempertingkatkan sifat kimia tanah terpilih secara bererti melalui kesan perbaikan Zeolit Klinoptilolit. Penggunaan Zeolit Klinoptilolit telah mengurangkan kehilangan larutlesap Ca dan Mg, justeru itu, keputusan yang diperolehi dalam kajian ini tanpa mengira rawatan adalah sama. Ketersediaan N, P, K, dan Fe dalam tanah berkurangan secara bererti dengan aplikasi Zeolit Klinoptilolit ekoran pengurangan baja 25%. Seperti yang dibuktikan dalam dua kitaran penanaman jagung, penggunaan Zeolit Klinoptilolit dalam bidang pertanian adalah bermanfaat memandangkan ianya boleh digunakan untuk mengurangkan penggunaan baja N, P, dan K yang tidak

seimbang dalam penanaman jagung (*Zea mays* L.) dan tanaman lain yang ditanam di tanah berasid. Selain itu, ianya mampu mengurangkan pencemaran alam sekitar disebabkan oleh penggunaan baja kimia yang berlebihan dan pergerakan unsur-unsur toksik. Analisis daya maju ekonomi bagi Zeolit Klinoptilolit dalam penanaman jagung mendedahkan bahawa jumlah kos pengeluaran berkurang dengan penggunaan Zeolit Klinoptilolit. Nisbah kos-faedah untuk kadar baja yang disyorkan adalah 1.69 manakala dengan penggunaan Zeolit Klinoptilolit manfaat nisbah-kos meningkat (1.83-1.84). Oleh itu, penggunaan Zeolit Klinoptilolit dengan jumlah baja yang dikurangkan didapati berdaya maju dari segi ekonomi kerana ianya bukan sahaja memberi pulangan yang lebih tinggi tetapi juga dapat memastikan kemampanan produktiviti pertanian dan kesuburan tanah.



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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment 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

AI-P	Aluminium bound P
BC	Buffering capacity
Ca-P	Calcium bound P
CEC	Cation exchange capacity
DAI	Days after incubation
DAP	Days after planting
DAS	Days after sowing
Fe-P	Iron bound P
Occl-P	Occluded P
ОМ	Organic matter
Sol-P	Loosely soluble P

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CHAPTER 1

INTRODUCTION

1.1 Background and Problem Statement

Phosphorus plays an important role in plant metabolic function as it is one of the essential nutrients required for lucrative crops production. Its deficiency in soils does not only limit nitrogen (N) uptake but it also leads to poor yield of crops (Bundy *et al.,* 2005). Phosphorus supplied in soluble form is rapidly converted to less soluble form by adsorption and precipitation thus, rendering it unavailable for crops uptake.

In acidic soil of the tropics, P is either inherently low or is fixed in forms that are unavailable to crops. Metal oxides and hydroxides such as AI and Fe which are dominant in highly weathered soils of the tropics fix large amounts of soluble P (Wilson et al., 2004). Thus, large amount of P fertilizers are applied to saturate the capacity for P sorption and to ensure P availability for crops uptake. However, this practice in agriculture is neither economical nor environmentally friendly. For example, excessive and inefficient use of P fertilizers endanger water bodies as P can be lost through soil erosion (Zhou and Zhu, 2003) and also leaching to cause eutrophication (Ruban, 1999). Besides, P which originates from non-renewable resources are limited in reserves (Bondre, 2011; Filippelli, 2011; Gilbert, 2009). The responses to P scarcity may include increased cost, more efficient P use, and P recovery and re-use. Moreover, prolonged use of chemical fertilizers in agriculture degrades soil fertility. Therefore, an effective management of P fertilizers will promote not only increase of crops yield but it will also reduce costs of production, mitigate environmental pollution besides conserving finite P deposit.

In Malaysia, the agricultural sector is the third contributor to the Malaysian economic growth. Approximately 90% of chemical fertilizers is required to sustain the agricultural sector of Malaysia. The fertilizer import bills were USD\$ 209 million and USD\$ 319 million in 2009 and 2013, respectively (FAOSTAT, 2014). The fertilizer import bill of Malaysia is estimated to increase yearly. The use of rock phosphates is preferable because they are cheaper and their dissolution is favorable in acidic condition instead of highly soluble P fertilizers such as Triple superphosphate (Akande *et al.*, 2008; Nnadi and Haque, 1998). Besides, the use of soil amendments such as organic and inorganic materials has been incorporated in some of the agriculture practices. One of the soil amendments is natural zeolites.

Zeolites are natural aluminosilicates with an infinite three-dimensional crystal structure, a polyedric shape, with a great open cavity (Ajirloo *et al.,* 2013;

Ramesh *et al.*, 2011; Daković *et al.*, 2007). These abundant and cheap minerals are used in agriculture as soil conditioners, slow-release fertilizers, and agents for contaminated soils (Ming and Allen, 2001). Phosphorus release through the use of zeolites and rock phosphate (RP) had been demonstrated by exchange-induced dissolution system as (Allen *et al.*, 1993):

RP (rock phosphate) + NH_4^+ + zeolite \rightarrow Ca-zeolite + NH_4^+ + $H_2PO_4^-$

Clinoptilolite zeolite which is one of the most important natural zeolites is useful to retain nutrients such as NH_4^+ and K^+ . The cations selectivity of zeolites is in the order of $Cs^+ > Rb^+ > K^+ > NH_4^+ > Ba^{2+} > Sr > Na^+ > Ca^{2+} > Fe^{3+} > Al^{3+} > Mg^{2+} > Li^+$ (Mumpton, 1999). Crops uptake of cations from Clinoptilolite zeolite leads to vacant exchange sites to which Ca^{2+} are attracted to. This process lowers the activity of Ca^{2+} from soil solution thereby inducing dissolution of rock phosphates (RP) and also, producing ammonium ions as by-product (Pickering *et al.*, 2002; Allen *et al.*, 1995; Barbarick *et al.*, 1993; Lai and Eberl, 1986).

Zeolites are not easily degraded Over time and because of this, they remain in soils to improve retention of nutrients and control nutrient release for crop use (Ramesh *et al.*, 2010; Eberl, 1993; Zelazny *et al.*, 1977). Amelioration of soil pH due to Clinoptilolite zeolite catalytic ability as an example could reduce soil acidity and concentration of exchangeable AI and Fe. Hence, reducing P fixation in acid soils, ensure efficient use of nutrients through timely release of nutrients for optimum crop production. Perhaps, in long term application of zeolites, soil properties could be rejuvenated to sustain crop productivity. Although zeolites have been extensively used in agriculture, there is dearth of information on the use of zeolites) on P sorption and fixation in highly weathered tropical soils not to mention reduction of N, P, and K fertilizers use in agriculture.

1.2 Objectives

The general objective of this study was to improve P use efficiency and to also reduce amount of fertilizers (N, P, and K) use by amending chemical fertilizers with Clinoptilolite zeolite in an acid soil (Bekenu series, Typic Paleudult). Thus, this study was carried out to determine the:

- 1. Effects of amending different rate of phosphate fertilizers (Triple superphosphate, Egypt rock phosphate, and Christmas Island rock phosphate) with Clinoptilolite zeolite on phosphorus dissolution and selected soil chemical properties.
- 2. Effects of amending phosphorus fertilizers with Clinoptilolite zeolite on dry matter production, nutrients concentration, nutrients uptake and use efficiency, and yield of *Zea mays* L. cultivated on an acidic soil.
- 3. Economic viability of amending phosphorus fertilizers with Clinoptilolite zeolite in the fertilization program of *Zea mays* L. cultivated on an acidic soil.

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BIODATA OF STUDENT

Nur Aainaa binti Hasbullah, born in 5th January, 1988 in Kuala Lumpur. She received her primary education in Sekolah Rendah Kebangsaan Ampang in 1995. She persued her secondary education in Sekolah Menengah Sains Hulu Selangor in 2000 and entered Malacca Matriculation College in 2005. In the following year, she continued her higher education at Universiti Putra Malaysia Bintulu Campus and graduated with Bachelor of Science Bioindustry (Honours First Class) in 2010. Afterwards, she was enrolled in degree of Doctor of Philosophy in Agronomy in 2011. During her postgraduate studies, she published three journal articles, attended Soil Science Conference of Malaysia in April, 2014 (won best poster award). In June, 2014 she attended the 20th World Congress of Soil Science in Jeju, Korea and her abstract won the Travel Grant for Young Scientist of Developing Country. Her thesis has been converted to a book which is being processed for publication.

LIST OF PUBLICATIONS

Published paper

- Hasbullah Nur Aainaa, Osumanu Haruna Ahmed, Susilawati Kasim, And Nik Muhamad Ab. Majid. 2014. Use of Clinoptilolite zeolite on selected soil Chemical Properties, Dry Matter Production, Nutrients Uptake and Use Efficiency of *Zea mays* L. cultivated on An Acid Soil. *International Journal of Agriculture Research*, 9: 136-148.
- Hasbullah Nur Aainaa, Osumanu Haruna Ahmed, Susilawati Kasim, And Nik Muhamad Ab. Majid. 2014. Use of Clinoptilolite zeolite to reduce Christmas Island Rock Phosphate use in *Zea mays* Cultivation on an Acid Soil. *International Journal of Soil Science*, 9: 55-66.
- Hasbullah Nur Aainaa, Osumanu Haruna Ahmed, Susilawati Kasim, And Nik Muhamad Ab. Majid. 2015. Reducing Egypt Rock Phosphate use in Zea mays Cultivation on an Acid Soil Using Clinoptilolite Zeolite. Sustainable Agriculture Research, 4(1): 56–66.

Presented paper

- Hasbullah Nur Aainaa, Osumanu Haruna Ahmed, Susilawati Kasim, And Nik Muhamad Ab, Majid. 2014. Reducing Egypt Rock Phosphate use in *Zea mays* Cultivation on an Acid Soil Using Clinoptilolite Zeolite. Presented at the 20th world congress of soil science, Jeju, South Korea. 8 – 13 June, 2014.
- Hasbullah Nur Aainaa, Osumanu Haruna Ahmed, Susilawati Kasim, And Nik Muhamad Ab. Majid. 2014. Reducing Christmas Island Rock Phosphate use in Zea mays Cultivation on an Acid Soil using Humic Acids. Presented at Soil Science Conference of Malaysia 2014. 8 - 10 April, 2014.

LIST OF AWARDS

- Silver Medal, "Biological agriculture to improve crops productivity without polluting the environment". Invention & Innovation Awards 2016, Malaysia Technology Expo 2016, Kuala Lumpur, Malaysia.
- Young scientist travel grant, "Reducing Egypt rock phosphate use in Zea mays cultivation on an acid soil using clinoptilolite Clinoptilolite zeolite". The 20th World Congress of Soil Science (20WCSS, 2014), Jeju Island, South Korea.
- Excellent Poster Presentation, "Reducing Christmas Island rock phosphate use in Zea mays cultivation on an acid soil using humic acids". Soil Science Conference of Malaysia 2014, Putra Palace Hotel, Kangar Perlis.