UNIVERSITI PUTRA MALAYSIA

***Acacia mangium* WILLD. AND *Melaleuca cajuputi* POWELL AS POTENTIAL HEAVY METAL ACCUMULATORS IN SEWAGE SLUDGE-CONTAMINATED SOILS**

NIK MOHD. SHIBLI BIN NIK JAAFAR

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By

NIK MOHD. SHIBLI BIN NIK JAAFAR

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Chairman: Professor Dato' Nik Muhamad Nik Ab Majid, PhD

Faculty : Forestry

Waste disposal in Malaysia faces serious challenges and has caused soil pollution. There are currently over 6 million cubic meters of sewage sludge in Malaysia requiring safe disposal. Phytoremediation is the use of plants to remediate polluted soils and this approach has not been practised in Malaysia. This study aimed to assess the potential and viability of two tropical timber species to remediate heavy metal contaminated soil. A greenhouse and field experiments were conducted to evaluate *Acacia mangium* Willd and *Melaleuca cajuputi* Powell to extract heavy metals namely Zinc (Zn), Copper (Cu) and Cadmium (Cd) from sewage sludge contaminated soils.

Results showed that under greenhouse conditions, both species are tolerant to Zn, Cu and Cd and could accumulate high concentrations of these elements especially in the roots. The leaves of *A. mangium* accumulated 156.96mg/kg of Zn and 135.20mg/kg in *M. cajuputi* leaves. In the roots, the concentrations were 266.25mg/kg and 137.19mg/kg, respectively. Accumulation of Zn in the stems of *A. mangium* was 102.24mg/kg and 107.50mg/kg for *M. cajuputi.* Accumulation of Cu in the leaves of *A. mangium* was higher (20.74mg/kg) than in *M. cajuputi* (9.53mg/kg) and in the roots it was 25.29mg/kg and 20.85mg/kg, respectively. The stems of *M. cajuputi* accumulated more Cu (19.27mg/kg) than *A. mangium* (14.01mg/kg). The accumulation of Cd in the stems and roots were greater in *M. cajuputi* with concentrations of 1.78mg/kg and 2.05mg/kg, compared in *A. mangium* at 1.51 mg/kg and 1.66 mg/kg, respectively. Accumulation of Cd in the leaves of *A. mangium* was slightly higher (1.93mg/kg) than *M. cajuputi* (1.66mg/kg).

The study under field conditions shows that stems of *A. mangium* accumulated up to 130.0mg/kg of Zn and in the leaves it was up to 100.0 mg/kg. Accumulation of Cu by *A. mangium* in the stems was about 17.0mg/kg and in the leaves was about 22.0mg/kg. The accumulation of Cd in the stems was 1.23 - 1.62mg/kg. However, Cd accumulation in the *A. mangium* leaves was 0.3 - 1.6mg/kg. Accumulation of Zn in *M. cajuputi* leaves was higher (80 - 100.0mg/kg) than in the stems (60 - 70.0mg/kg). The stems of *M. cajuputi* accumulated 9.0 - 22.0mg/kg Cu and in the leaves it was 4.0 - 15.0mg/kg. While Cd accumulation in the stems was 0.30 - 0.45mg/kg and in leaves it was 0.25 - 0.4mg/kg.

In the greenhouse experiment, both species show low ability to accumulate Cu and Cd as reflected by low BCF (BCF ≤ 1) but translocate high amounts of all three elements to the shoots as shown by high TF (TF ≥ 1). On the other hand, in the field experiment, *A. mangium* showed good ability (BCF ≥ 1) for extracting Zn and Cd but only Zn for *M. cajuputi*. However, *M. cajuputi* stems accumulated more heavy metals compared to the leaves indicating that the heavy metals can be stored for a longer period.

Phytoextraction efficiency of *A. mangium* was better than *M. cajuputi* as the biomass generated in the field experiment after 12 months was about 72 tonnes/ha compared to only 4 tonnes/ha in case of *M. cajuputi*. It was estimated that *A. mangium* is able to extract 16.49kg/ha Zn, 2.80kg/ha Cu and 0.12kg/ha Cd per year whereas *M. cajuputi* could only extract 12.91kg/ha/year, 2.65kg/ha/year and 0.06kg/ha/year of the three heavy metals, respectively.

The trees also affect mobility of Zn, Cu and Cd in the soil as shown by high fractions of the ionic and reducible forms of these metals at the beginning. The mobility reduced at the end of the study where these two fractions were reduced to about 2% from 69% for Cu and about 23% from 53% for Cd but Zn remained the same. Principal component analysis of the soil chemical properties shows humic acids played an important role (1st component) in heavy metal stabilization in the soil after planting and the effects of pH and CEC on metal mobility were reduced (2nd component) thus lowering the risk of these elements from spreading.

The cost-effective economic evaluation of *A. mangium* in this study for a 10-year rotation period was estimated to cost only MYR 231,800.00/ha compared to MYR 3 million/ha using the conventional soil remediation techniques. Within this period, over 35, 800 tonnes of carbon/ha was produced that could generate returns of about MYR 1.9 million/ha. Timber production was estimated at 25,300m3/ha and could generate returns of about MYR 6.33 million/ha. Hence, this study shows that phytoremediation using trees species is economically viable for disposal of sewage sludge.

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

Acacia mangium WILLD DAN _Melaleuca cajuputi_ POWELL SEBAGAI PENUMPUK LOGAM BERAT DARI TANAH YANG DICEMARI OLEH ENAPCEMAR KUMBAHAN

Oleh

NIK MOHD. SHIBLI BIN NIK JAAFAR

Jun 2014

Pengerusi: Professor Dato' Nik Muhamad Nik Ab. Majid, PhD

Fakulti : Perhutanan

Proses pelupusan sisa di Malaysia telah menyebabkan pencemaran tanah dan menjadi suatu cabaran yang serius. Pada masa ini di Semenanjung Malaysia terdapat lebih dari 6 juta meter padu enapcemar kumbahan yang perlu dilupuskan dengan selamat dan lebih sejuta hektar tanah terdegradasi yang perlu dipulihkan. Fitoremediasi (Phytoremediation) ialah teknik memulihkan tanah tercemar menggunakan tumbuh-tumbuhan dan kaedah ini belum digunapakai di Malaysia. Kajian ini bertujuan untuk menilai potensi dua spesies tumbuhan berkayu tropika untuk fitoremediasi dan kesesuaian ekonomi penggunaan kaedah ini untuk merawat tanah yang tercemar dengan logam berat.

Satu kajian di dalam rumah hijau dan di lapangan telah dijalankan untuk menilai kemampuan *Acacia mangium* dan *Melaleuca cajuputi* untuk mengekstrak logam berat iaitu Zink (Zn), Tembaga (Cu) dan Kadmium (Cd) daripada tanah yang dicemari dengan enapcemar kumbahan. Dalam eksperimen rumah hijau, empat tahap media rawatan campuran enapcemar telah disediakan iaitu Kawalan (0%), 30%, 50% dan 70% campuran enapcemar. Di lapangan pula, spesies ini ditanam di dalam barisan di sebuah tapak pelupusan enapcemar kumbahan.

Hasil kajian rumah hijau menunjukkan kedua-dua spesies mempunyai ciri-ciri tumbuhan yang toleran kepada logam berat dan mengelakkan keracunan logam berat dengan mengumpulnya di dalam akar di dalam jumlah yang tinggi. Daun *A. mangium* mengumpul 156.96mg/kg Zn dan135.20mg/kg Zn bagi daun *M. cajuputi*. Di dalam akar, kepekatan masing-masing adalah 266.25mg/kg and

137.19mg/kg. Manakala penimbunan Zn di dalam batang *A. mangium* adalah 102.24mg/kg dan 107.50mg/kg bagi *M. cajuputi*. Penimbunan Tembaga di dalam daun *A. mangium* adalah lebih tinggi (20.74mg/kg) berbanding di dalam daun *M. cajuputi* (9.53mg/kg) serta di dalam akar masing-masing adalah 25.29mg/kg dan 20.85mg/kg. Batang *M. cajuputi* menimbunkan banyak Tembaga (19.27mg/kg) berbanding *A. mangium* (14.01mg/kg).Penimbunan Kadmium pula di dalam batang dan akar *M. cajuputi* adalah lebih tinggi dengan kepekatan masing-masing adalah1.78mg/kg dan 2.05mg/kg, berbanding *A. mangium* yang hanya berkepekatan masing-masing 1.51mg/kg dan 1.66mg/kg. Penimbunan Kadmium di dalam daun *A. mangium* tinggi sedikit (1.93mg/kg) berbanding *M. cajuputi* (1.66mg/kg).

Hasil kajian di lapangan menunjukkan batang *A. mangium* menimbunkan sehingga 130.0mg/kg Zink dan di dalam daun penimbunannya sehingga 100.0 mg/kg. Penimbunan Tembaga oleh *A. mangium* di dalam batang adalah sekitar 17.0mg/kg dan di dalam daun pula sekitar 22.0mg/kg. Penimbunan Kadmium di dalam batang pula 1.23 - 1.62mg/kg. Manakala penimbunan Kadmium di dalam daun *A. mangium* adalah 0.3 - 1.6mg/kg. Penimbunan Zink di dalam daun *M. cajuputi* adalah lebih tinggi (80 - 100.0mg/kg) dari batangnya (60 - 70.0mg/kg). Manakala Tembaga di dalam batang *M. cajuputi* adalah 9.0 - 22.0mg/kg, di dalam daun adalah 4.0 - 15.0mg/kg dan Kadmium di dalam batang adalah 0.30 - 0.45mg/kg dan di dalam daun adalah0.25 - 0.4mg/kg.

Hasil kajian lapangan pula menunjukkan batang *A. mangium* menimbunkan Zink sehingga 130.0mg/kg manakala di dalam daun pula hingga 100.0 mg/kg.Penimbunan Tembaga oleh *A. mangium* di dalam batang adalah sekitar 17.0mg/kg dan di dalam daun pula sekitar 22.0mg/kg.Penimbunan Kadmium di dalam batang pula adalah 1.23 - 1.62mg/kg. Namun, Kadmium di dalam daun *A. mangium* adalah 0.3 - 1.6mg/kg. Penimbunan Zink oleh daun *M. cajuputi* adalah lebih tinggi (80 - 100.0mg/kg) berbanding batangnya (60 - 70.0mg/kg). Manakala Tembaga di dalam batang *M. cajuputi* adalah 9.0 - 22.0mg/kg dan daun adalah 4.0 - 15.0mg/kg. Sementara Kadmium di dalam batang adalah 0.30 - 0.45mg/kg dan 0.25 - 0.4mg/kg di dalam daun.

Kajian rumah hijau menunjukkan kedua spesis yang dikaji mempunyai kemampuan menumpuk Tembaga dan Kadmium yang rendah yang ditunjukkan oleh nilai BCF yang rendah (BCF ≤ 1) tapi mampu memindahkan jumlah yang banyak dari akar ke pucuk untuk kesemua elemen yang ditunjukkan dengan nilai TF yang tinggi (TF ≥ 1). Walaupun begitu, kajian lapangan berlaku perkara sebaliknya di mana *A. mangium* menujukkan kemampuan menimbun yang tinggi untuk Zink dan Kadmium dengan nilai BCF ≥ 1 kecuali *M. cajuputi* yang hanyamenunjukkankemampuanmenimbunkanhanya Zink. Pun begitu, batang*M. Cajuputi* dilihat mampu menumpukkan lebih banyak logam berat berbanding daunnya yang memberikan gambaran logam berat boleh disimpan di batang pokok ini lebih lama.

Kecekapan ekstraksi *A. mangium* adalah lebih baik berbanding *M. cajuputi* dimana jisim biologi yang dihasilkan dalam tempoh 12 bulan dianggarkan masing-masing berjumlah 72 tan/ha berbanding hanya 4 tan/ha. Dianggarkan bahawa *A. mangium* mampu untuk mengekstrak 16.49kg/ha Zink, 2.80kg/ha Tembaga dan 0.12kg/ha Kadmium setahun manakala *M. cajuputi* hanya boleh mengekstrak masing-masing 12.91kg/ha/tahun, 2.65kg/ha/tahun dan 0.06kg/ha/tahun.

Fitoremediasi juga didapati telah menghadkan pergerakan logam berat di dalam tanah. Pergerakan Zn, Cu dan Cd di awal kajian adalah tinggi di mana peratus logam berat bentuk ionic atau terturun adalah tinggi. Mobiliti elemen ini telah berkurang diakhir tempoh kajian di mana bagi Tembaga (Cu) hanya tinggal sekitar 2% dari 69% di awalnya dan sekitar 23% dari 53% untuk Kadmium. Walaubagaimanapun Zink masih kekal mudah tersebar.Analisis komponen utama telah dibuat untuk sifat-sifat kimia tanah yang mempengaruhi pergerakan logam berat menunjukkan keputusan di mana asid humik memainkan peranan yang penting (komponen pertama) dalam penstabilan logam berat selepas ditanam pokok ujian manakala kesan keasidan (pH) serta Kapasiti Tukarganti Kation (CEC) telah dikurangkan (komponen kedua) menunjukkan risiko logam berat untuk merebak di dalam tanah telah berkurangan.

Penilaian ekonomi fitoremediasi menunjukkan bahawa teknik pemulihan ini berdaya maju. Kajian ini menganggarkan di dalam tempoh kitaran 10 tahun ia memerlukan kos keseluruhan MYR 231,800.00/ha berbanding dengan teknik konvensional dengan kos sekitar RM 3 juta/ha. Di dalam tempoh masa yang sama lebih 35, 800 tan karbon/ha mampu dihasilkan dan boleh menjana pulangan kira-kira RM 1.9 juta/ha. Satu lagi sumber pendapatan adalah kayu yang boleh menghasilkan kira-kira 25,300 m^3dan boleh menjana pulangan kira-kira RM 6.33 juta/ha.

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I certify that a Thesis Examination Committee has met on 25 June 2014 to conduct the final examination of Nik Mohd Shibli bin Nik Jaafar on his thesis entitled "*Acacia mangium* Willd. and *Melaleuca cajuputi* Powell as Potential Heavy Metal Accumulators in Sewage Sludge-Contaminated Soils" 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 Doctor of Philosophy.

Members of the Thesis Examination Committee were as follows:

Ahmad Ainuddin b Nuruddin, PhD
Associate Professor
Faculty of Forestry
Universiti Putra Malaysia
(Chairman)

Osumanu Haruna Ahmed, PhD
Associate Professor
Faculty of Agriculture and Food Sciences
Universiti Putra Malaysia Kampus Bintulu
(Internal Examiner)

Ahmad b Ismail, PhD
Professor
Faculty of Science
Universiti Putra Malaysia
(Internal Examiner)

Muhammad Abi, PhD
Professor
Faculty of Agricultural Sciences & Technology
Bahauddin Zakariya University
(External Examiner)

ZULKARNAIN ZAINAL, PhD
Professor and Deputy Dean
School of Graduate Studies
Universiti Putra Malaysia

Date: 23 January 2015

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

Dato' Nik Muhamad Nik Ab Majid, PhD
Professor
Institute of Tropical Forestry and Forest Product,
Universiti Putra Malaysia
(Chairman)

Noor Azhar Mohamed Shazili, PhD
Professor
School of Marine Science and Environment,
Universiti Malaysia Terengganu
(Member)

Arifin Abdu, PhD
Associate Professor
Faculty of Forestry,
Universiti Putra Malaysia
(Member)

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Professor and Dean
School of Graduate Studies
Universiti Putra Malaysia

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Signature

Name of Chairman of Supervisory Committee: **Dato' Nik Muhamad Nik Ab Majid, PhD.**
(Professor)
Faculty of Forestry,
Universiti Putra Malaysia

Signature

Name of Member of Supervisory Committee: **Noor Azhar Mohamed Shazili, PhD.**
(Professor)
School of Marine Science and Environment,
Universiti Malaysia Terengganu

Signature

Name of member of Supervisory Committee: **Arifin Abdu, PhD.**
(Associate Professor)
Faculty of Forestry
Universiti Putra Malaysia

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LIST OF ABBREVIATIONS, UNITS AND SYMBOLS

AAS	Atomic Absorption Spectrophotometer
ANOVA	Analysis of variance
BCF	Bio-concentration factor
CEC	Cation Exchange Capacity
DOA	Department of Agriculture, Malaysia
F1	Easily, Freely, Leachable and Exchangeable Fraction
F2	Acid Reduced Fraction
F3	Organic Bound Fraction
F4	Residual Fraction
HCl	Hydrochloric acid
ha	Hectare
IWK	Indah Water KonsortiumSdnBhd
kg/ha	Kilogramme per hectare
mg/kg	Miligramme per kilogramme
MYR	Malaysian Ringgit
NaOH	Sodium hydroxides
NIST	National Institute of Standard and Technology
POPs	Persistent Organic Pollutants
PPCPs	Pharmaceutical and Personal Care Products
SRM	Standard Reference Material
SS	Sewage sludge
t/ha	Metric tons per hectare
T1	Treatment 1
T2	Treatment 2
T3	Treatment 3

T4	Treatment 4
TF	Translocation factor
TN	Total nitrogen
USD	United States Dollar
USDA	United States Department of Agriculture
USEPA	United States Environment Protection Agency
v/v	Volume to volume ratio
€	Euro Dollar

CHAPTER 1

INTRODUCTION

1.1 Study Background

Soil is an important component of life on earth. It is crucial in various nutrient and hydrological cycles and it is the basis of food chain. Almost all food sources originate from primary producers which grow on soil with few exceptions from aquatic sources (Bridges and Van Baren, 1997). The increase in world population put pressure on soil resources to continuously provide food (Khanif, 2010). Currently world population exceeds 6 billion and predicted to reach 8 billion by the year 2020. This is an unavoidable fact that per capita of arable land is decreasing steadily. It has been reported that arable land has being reduced to 0.22ha per person (Bridges and Van Baren, 1997;Khanif, 2010).

Land degradation and environmental pollution will be more serious as population increases. Innovative science and technology are necessary to reduce land degradation and increase agriculture production. Land degradation will remain an important global issue in the 21st century because of its negative impact on crop productivity, environment, food security and quality of life (Eswaran *et al*., 2001). Soil or land degradation is defined as soil that has lost its functions or its ability to support life. It is a result of mismanagement, over exploitation and mismatch of land use with soil quality. Major stresses on degraded soil are erosion, nutrient loss, acidity, salination, destruction of soil structure and loss of organic matter (United States Department of Agriculture, 2007).

Bridges and van Baren (1997) stated that soil degradation can be categorized into three types; chemical, physical and biological. Physical degradation is mostly due to the weight and size of machineries used to cultivate the soil. Soil compaction affects soil porosity and infiltration capacity and eventually restricts root penetration. Compaction also limits oxygen penetration into soil and inhibits aerobic processes of soil microbes and in turn may adversely influence soil pH and nutrient cycling processes.

Soil chemical degradation relates to nutrient depletion or soil contamination. Nutrient depletion occurs naturally by erosion, leaching and uptake by plants. Nutrient depletion or low soil fertility is the main constraint in agriculture production. Fertilizers are being used excessively to increase agricultural yield. The desire for mobility, goods and services has led to industrialization. Industries are generating hazardous wastes that pollute the environment and causing health problems (Chaaban, 2001). Langergraber and Muelegger (2005) stated that over two billion people around the world mainly in developing countries still have no access to adequate sanitation services and over 90% of sewage from these countries is discharged without proper treatment.

In addition, the increasing amount of solid wastes from urban areas is no longer suitable to be treated through conventional land disposal method. Soil capacity to store various chemicals and other toxic elements is limited. Soils are dynamic and have different resilience (Bridges and Van Baren, 1997, Khanif, 2010). Extreme changes in soil physical and chemical characteristics cause the chemicals in the soil to be unleash and this is known as “chemical time bomb” (Cappuyns *et al*., 2006; van der Wal *et al*., 2007).

Degraded soil has resulted in negative ecological consequences such as poor air and water quality, eutrophication of fresh water bodies, algal blooms in coastal waters, global warming, acid rain, increase of diseases and biodiversity loss (Zhao *et al*., 2006; Ayoub, 1999). Soil degradation affects the soil microbes including soil fauna such as earthworms that makes the soil functional. Environmental pollution is no longer a localized problem as researchers have found that in recent years, a number of toxic accidents, acid rain and poor waste management in developing countries have caused pollution to spread beyond its source point boundaries (Scanlon, 2001).

1.2 Problem Statement

Soil degradation is a worldwide problem. However unlike in arid and semi arid regions of the world, soil degradation in Malaysia is categorized as moderate to low risk because of the tropical climate (van Lynden and Oldeman, 1997). Soil degradation in arid and semi-arid is attributed to lack of rainfall but in Malaysia, it badly affects neighbouring areas resulting in erosion and silting. Abdullah and Hezri (2008) reported agriculture as a major landuse in Malaysia utilizing over 46% of land cover and is increasing annually through conversion of forested areas mainly for palm oil. This has resulted in serious environmental consequences mainly loss of biodiversity, geohazards incidences and environmental pollution.

Accurate information on land degradation in Malaysia is not readily available. However according to Forestry Department of Peninsular Malaysia, (FDPM) and Department of Agriculture (DOA), it is estimated that more than 20 million hectares or 60% of total land area are categorized as environmentally fragile and degraded. The most degraded soil is mined land, covering approximately 200,000 ha in Peninsular Malaysia. In addition, approximately 2.7 million ha of the total land area are under shifting cultivation, especially in Sarawak and at various degrees of degradation (DOA, 2006).

According to the Malaysian Department of Statistics (2009), 62% of the country's population lives in urban areas compared only 26% in 1970. Khanif (2010) stated that the Malaysian population is projected to reach 30 million by 2020. This will lead to land use change and perhaps encroachment into the environmentally fragile areas. This encroachment has already occurred where 46% of peat areas in Malaysia or approximately 432,520ha peat swamps have been drained and converted mainly for oil palm cultivation (DOA, 2006).

Utilization of environmentally fragile areas and urbanization also lead to pollution and other environmental problems. According to Hassan *et al*., (1999), there are 230 authorized landfill sites in Peninsular Malaysia where 74% are causing serious problems particularly groundwater pollution. National Sewerage Management Company, Indah Water Konsortium Sendirian Berhad (IWK) reported that sewage sludge production in Peninsular Malaysia has increased 1 million m^3 in a 10-year period from 5 to 6 million m^3 in 1997 to 2007 (IWK, 2007). The sewage sludge eventually will end up in landfills for disposal. Hence, waste disposal problems compounded with agriculture expansion are foreseeable facts that soil degradation is a significant challenge for sustainable development in Malaysia.

1.3 Significance of Study

Projected population of Malaysia is 30 million in 2020 with over 62% living in urban areas and this will increase demand for land. There is also an urgent need to manage waste and sewage disposal problems and at the same time rehabilitate degraded land for future use. The use of plants to remediate polluted soil or phytoremediation is still in infancy stage in Malaysia. Unlike conventional methods of remediating contaminated soil, phytoremediation provides opportunities to utilize plant biomass and other related secondary plant products for economic benefits.

1.4 Objectives

Studies on phytoremediation with timber species have not been widely reported. Therefore the general objective of this study was to assess the efficacy of timber tree species of high economic value to remediate soil contaminated with heavy metals from sewage sludge. The specific objectives are:

1. Evaluate ability of the species in accumulating heavy metals namely Zn, Cu and Cd and its phytoextraction efficiency.

2. Evaluate economic potential of phytoremediation using trees.

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