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Solid Waste Management

Land Application of Agricultural & Industrial By-products

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ABSTRACT

The generation of industrial wastes shows an increasing trend throughout the world including Malaysia. Recycling of these wastes or by-products on land is the most economically feasible. However, the presence of trace metal(loid)s is an issue of concern. Therefore, these by-products need to be assessed in terms of trace metal(loid)s content and their bioavailability before they can be land-applied. Bioavailability of trace metal(loid)s in the soil system can be transferred to the food chain via plant uptake and leaching to ground water or transported via surface runoffs to water bodies. Thus, the ultimate goal of remediation is to reduce the transfer of trace metal(loid)s to the food chain. One of the remediation method recommended is the *in-situ* immobilization technique. In this technique, the remediation of trace metal(loids-rich or contaminated soils will be through manipulating their bioavailability by using a range of soil amendments such as liming materials, organic materials or biosolids, phosphate compounds and metal oxides (Fe-rich materials). The soil chemical processes exploited to immobilize metal(loid)s *in-situ* are the adsorption and precipitation processes.

INTRODUCTION

Just like many industrialized countries in the world, Malaysia is also facing problems of the disposal of industrial waste. Instead of leaving the waste as stack within the vicinity of the industrial plant or sending the waste to hazardous waste landfill which is costly, the waste should be assessed for its suitability for land application. Land application or recycling the wastes on land is perhaps the most economical or cost-effective in terms of utilization.

The main constraint of land application of industrial by-products is the trace metal(loid)s present in the by-products. Thus, most of the industrial by-products are categorized as scheduled waste. Agricultural by-products, on the other hand, tend to have lower trace metal(loid)s content and the issue of metal(loid)s content rarely arise. When the industrial by-products are applied to agricultural land, the trace metal(loid)s can be taken up by the plant and affect the quality of the produce in terms of trace metal(loid)s content. Trace metal content in agricultural produce is one of the food safety issue which is gaining prominence.

Unlike organic contaminants, metal(loid)s do not undergo microbial or chemical degradation and persist for a long time unless transported out of the site. Also, trace metal(loid)s can exist naturally in the soil system from the minerals present in the parent materials from which the soil was derived and introduced to the soil system via anthropogenic activities such as agriculture inputs. Therefore, there is a need to establish a baseline or background levels of trace metal(loid)s in the soil system

The trace metal(loid)s of concern in the food chain are As, Cd, Co, Cr, Cu, Hg, Mn, Mo Ni, Fe and Zn. Of these, Cd is of most concern because its transfer from soils to the edible portions of agricultural food crops is significantly greater than for other elements and can be hazardous to human health at very low level.

According to WHO, the maximum daily requirement for human intake is 3 mg per kg of body weight. The main pathway by which trace metal(loid)s end up in the human body is via the food chain. One of the pathway is through consumption of plants with high levels of these metal(loid)s.

After the establishment of the soil baseline or background value, the next phase of research is to assess the suitability of the industrial by-products for land application, especially agricultural land as these by-products can also contribute to plant nutrients content to the soil system, hence improve the fertility status of the soil. The environmental impact of these by-products application also need to be investigated due to the trace metal(loid)s content. The by-products that have been assessed this far are industrial biosolids such as sewage sludge, paper mill sludge and coal fly ash, and the mineral by-products such as red gypsum and water treatment residues.

The next phase of research is to assess the capability of the by-products to remediate soils rich or contaminated with trace metal(loid)s. Although these by-products contain certain levels of trace metal(loid)s, they do have unique characteristics which enable them to inhibit metal(loid)s mobility in the soil system. The focus of the research is on *in-situ* immobilization technique of metal(loid)s in the soil system. The by-products utilized can be classified as liming materials, metal oxides, Fe-rich minerals, phosphate compounds, organic compounds and biochar.

SOURCES OF TRACE METAL(LOID)S IN THE SOIL SYSTEM

In terrestrial ecosystems, the soil is the main repository of chemical contaminants. Metal(loid)s can reach the soil environment through both geogenic or pedogenic and anthropogenic processes. Most metal(loid)s occur naturally in the minerals present in the soil parent materials, mainly in the forms that are not readily available for plant uptake.

Geogenic or Pedogenic Sources

Most of the trace metal(loid)s occur in nature, the major source of which is weathering of soil parent materials. Infact, the majority of arsenic is derived from geogenic origin. As an example, weathering of basalt in the past thousands of years under the conditions prevailing in Malaysia had resulted in the formation of highly weathered soil known as the Kuantan Series soil (Tessens and Shamshuddin, 1983), which occupy 1 to 2 m above the bauxite layer. Metal(loids) such as As, Cd and Pb, especially Cr and Ni which is present in excess, are continuously being taken up by plants growing on this soil. The saprolite (a layer of rock materials mixed with soil or termed as C horizon of the soil) that contains bauxite would certainly have higher trace metal(loid)s content than the soil materials above it. In a study on heavy metals content in soils of Peninsular Malaysia grown with cocoa, it was found that Cu and Ni (Cu:36 -51 mg/kg; Ni: 20 -37 mg/kg) were relatively higher in soils derived from andesitic parent material compared to mean Cu concentration (16 mg/kg) and mean Ni concentration (14 mg/kg) in agricultural soils of Peninsular Malaysia (Fauziah et al., 2001). In their study, Mahsa et al. (2014) reported very high concentrations of three trace metal(loid)s, which are Cr, Ni and Co in serpentinite soils of Malaysia, with concentrations range of 2,427 – 27,863 mg/kg, 850- 4,753 mg/kg and 35-167 mg/kg, respectively.

Anthropogenic Sources

Anthropogenic trace metal(loid)s contamination of various environments is a persistent problem in industrial societies. Unlike pedogenic inputs, metal(loid)s added through anthropogenic inputs are typically more reactive and high in bioavailability (Adriano, 2001). These contaminants are non-degradable and they accumulate in the upper layers of soils. Therefore, soils constitute reservoirs of bioavailable metal(loid)s that can lead to bioaccumulation of toxic elements in the food chain, and induce perturbation of the ecosystem and adverse health effects. Examples of anthropogenic activities which are related to agriculture and responsible for trace metal(loid)s enrichment of agricultural soils are application of P fertilizers, the heavy usage of poultry manure, especially in organic farming, and pesticides, especially copper-based fungicides.

Metal(loid)s Concentrations in Phosphate Rock Fertilizers

Malaysia does not have her own source of phosphate rock fertilizers and thus these fertilizers have to be imported. Malaysia import most of the PR fertilizers from the Middle East and these are mainly of the sedimentary type. The trace metal(loid) of concern in phosphate rock fertilizers is Cd. All phosphate rocks contain some level of Cd, with the concentration being dependent on the origin of the phosphate ore. The sedimentary phosphate rocks, sources of 80% of the P fertilizers on the global market, contain higher levels of Cd than are found in igneous (volcanic) phosphate rocks. Cadmium contamination of agricultural soils is of particular concern because it reaches the food chain through the regular use of the Cd-containing P fertilizers. The Table on the next page shows the Cd levels of some of the PRs available commercially.

Table 1 Metal(loid)s concentrations of some commercial phosphate rock

	Fe	Cd	Cu	Zn	Mn	Pb
	(mg/kg)					
China Phosphate Rock	2781.52 [1.30]	9.43 [0.50]	16.33 [1.26]	187.68 [0.19]	631.84 [0.62]	135.61 [8.32]
Morocco Phosphate Rock	1107.39 [3.01]	19.07 [2.35]	18.26 [2.31]	149.41 [0.88]	28.08 [0]	19.14 [8.32]
Jordan Phosphate Rock	1204.56 [1.31]	8.20 [0.58]	17.87 [0]	170.81 [0.47]	21.92 [0]	31.61 [13.58]
Christmas Island Phosphate Rock	2960.87 [4.99]	21.79 [0.50]	54.47 [1.94]	299.79 [0.47]	448.03 [23.58]	52.41 [8.32]
North Carolina Phosphate Rock	2276.09 [3.69]	31.42 [2.14]	18.26 [1.47]	344.74 [0.77]	32.08 [0.62]	19.14 [8.32]

(Das, 1994)

Trace Metal(loid)s Concentrations of Poultry Manure

The Malaysian vegetable growing farmers rely heavily on poultry manure for the vegetables production. The poultry manure can be a source of trace metal(loid)s as shown in Table 2. Even the organic farming practices are not spared from these heavy metals issue. Copper is one element of concern because some poultry farmers use copper as growth promoter in poultry units (Bolan et al., 2003).

Table 2 Selected trace metal(loid)s concentrations of layer and broiler manure

Manure		Zn	Cu	Cd	Pb	As
		(mg/kg)				
Layer	UPM	2448	254	0.60	5.46	-
	Selangor	467.2	12	0.18	2.99	-
Broiler	UPM	623.2	654	0.15	5.55	-
	Selangor	469.6	402	0.09	1.79	-
	Seremban	814	748	0.20	8.92	8.49
	Semenyih	955.6	586	0.16	5.89	6.23

(Unpublished data)

Trace Metal(loid)s Distribution of Agricultural Soils of Peninsular Malaysia

From our earlier work undertaken to study the heavy metals distribution in agricultural soils of Malaysia (Zarcinas et al., 2004), based on principal component analysis (PCA) shown on the next page (Figure 1), concentrations of Co, Ni, Pb and Zn in the soils were strongly correlated with soils concentrations of Al and Fe, which suggest evidence of background variations due to changes in soil mineralogy. Chromium was correlated with pH and EC,

Na, S and Ca suggesting association with acid sulphate soil and soil salinity components, while Hg was not correlated with any of these components, suggesting diffuse pollution by aerial deposition. Arsenic, Cd, Cu were strongly associated with aqua-regia soluble and available P, and organic matter, suggesting these metals are associated with agricultural inputs in agricultural fertilisers and soil organic amendments. This finding indicates metal(loid)s contamination to the majority of agricultural soils in Peninsular Malaysia is due to anthropogenic activities (possibly added in fertilizers, wastes, pesticides, effluents or atmospheric sources) that may pose risks to the environment or human health.

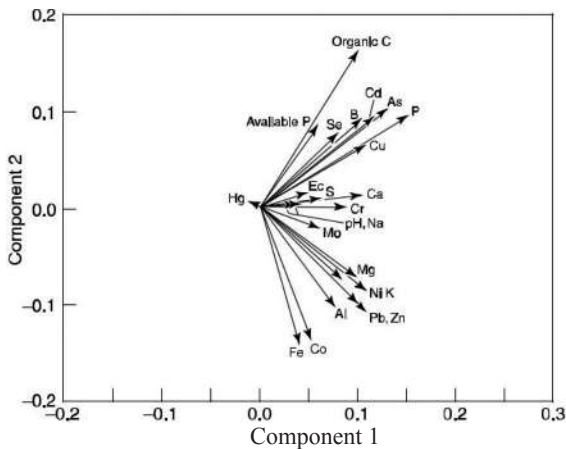


Figure 1 PCA of the trace metal(loid)s and fertility parameters for the Malaysian soils sampled in this survey

ESTABLISHMENT OF INVESTIGATION LEVELS OF SELECTED TRACE METAL(LOID)S IN MALAYSIAN SOILS

In a soil geochemical and concomitant plant survey to assess the extent of trace metal(loid)s contamination of soils and crops in Malaysia, the 95th percentile investigation levels of selected metal(loid)s (Table 3) were established (Zarcinas et al., 2004). This “95% protection level” was generated to establish a soil quality criterion to ensure protection of the terrestrial environment from the adverse effects of soil pollution. This metal(loid)s investigation levels will be used as a benchmark to decide when remediation should be conducted on the so-called trace metal(loid)s rich or contaminated soil or the by-products is suitable for land application where there are no guidelines on maximum permitted concentrations of trace metal(loid)s are available.

Table 3 The 95% ‘Investigation Levels’ determined for soils of Malaysia ($n = 241$ soils)

Element	Investigation level (mg kg ⁻¹)
As	60
Cd	0.30
Co	10
Cr	60
Cu	50
Hg	0.35
Ni	45
Pb	65
Zn	95

ASSESSMENT OF THE SUITABILITY OF BY-PRODUCTS TO BE LAND-APPLIED

Agriculture will never be sustainable as long as soil organic matter levels are on a multi-year downward trend. Thus, soil organic matter must be restored to near original levels. Utilization of biosolids can help build-up organic matter in the soil system. Many mineral industrial by-products, such as red gypsum, contain calcium and iron. Use of calcium helps build up the supply of soil organic matter in ways other than those for which calcium sources are usually added to soil. Calcium supplied as gypsum will be a major means for increasing the efficiency of accumulation of soil organic matter. The role of calcium in stabilizing soil organic matter needs more study. The mechanism that binds organic matter to clay particles in soil is polyvalent cations (Muneer and Oades, 1989). Efforts to increase levels of soil organic matter have overlooked this important phenomenon. Calcium is the most important cation for this purpose. Just how much calcium is necessary to bind organic matter to clay and how calcium relates to “slow” and passive soil organic matter need to be subjects for study. Iron on the other hand, is also a good bridging agent. Use of calcium and iron with organic matter could be the great secret for soil improvement.

The by-products that have been assessed this far are industrial biosolids such as sewage sludge, paper mill sludge and coal fly ash, and the mineral by-products such as red gypsum. Table 4 summarise the benefits and metal(loid)s of concern when these by-products are land applied.

Table 4 Benefits and limitations of by-products to be land-applied.

By-product	Benefit	Metal(loid)s of concern	References
Domestic sewage sludge	Increase soil organic carbon	Cu and Zn	Rosazlin et al., 2005
	Source of N		Rosazlin et al., 2006
Paper mill sludge	Increase soil organic carbon	No metal(loid)s of concern	Rosazlin et al., 2015a
	Source of N		Rosazlin et al., 2015b
			Rosazlin et al., 2015c
Coal fly ash	Source of Ca and Mg	B	Fauziah et al., 1999
	Pozzolanic material for slope stabilization		
Red gypsum	Source of Ca and S	Fe and Ti	Fauziah et al., 1996
	Reduce soil loss from erosion		

Sewage Sludge

The projected sludge generation for 2035 is estimated at 10 million m³ per year (wet wt. basis) with an average 2% solids content (Anonymous, 2010). Recycling of sewage sludge to agricultural land can be beneficial from the aspect of essential plant nutrients and organic matter supplement to the soil and plant system. However, sewage sludge also contains varying amounts of trace metal(loid)s which may pose a metal toxicity hazard to crops and consumers of these crops.

The sewage sludge research is of international and local interests. The research took off in 1996 until 2001 from the funding secured from the International Atomic Energy Agency (IAEA), Vienna, under FAO/IAEA Co-Ordinated Research Programme with the project title “The Use of Irradiated Sewage Sludge to Increase Soil Fertility, Crop Yields and to Preserve the Environment”. In this research, the ¹⁵N-isotope technique was used to determine percentage of N-derived from sewage sludge which was taken up by the corn plant (Fauziah et al., 2000; Rosenani et al., 2000). The availability of sludge N increased with the sludge application rate of up to 300% recommended N rate and then decreased with the higher rates (450 and 600% N recommended N rate) in both non-irradiated and irradiated sludge treatments. This is attributed to the inhibition effect of the sludge on crop growth at the higher rate, and thus lower total N uptake. The total sludge N uptake was 37 to 76% of the total N uptake.

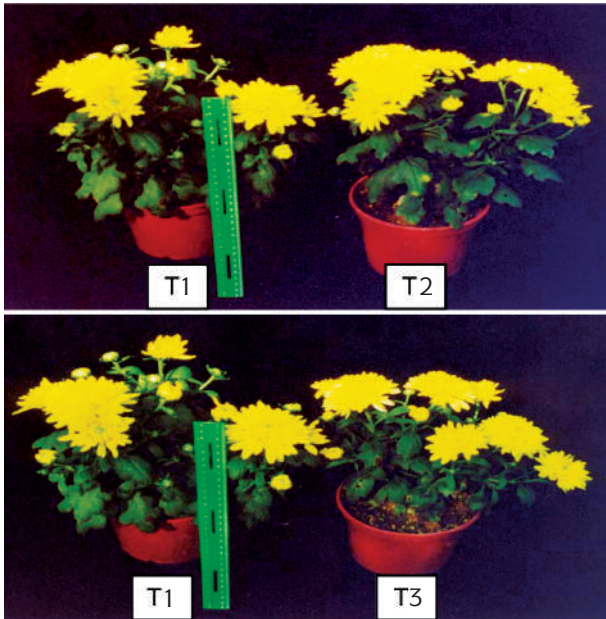
Another research project “Utilization of sewage sludge as fertilizer” funded by Indah Water Konsortiom (IWK) was conducted from 1999 until 2001. In the IWK funded research, a soil incubation study was conducted to investigate the potential use of sewage sludge produced by Indah Water Konsortium (IWK) as nitrogen

fertilizers for corn cultivation. In this study, the composition of sewage sludges and N- mineralization potential of a selected sludge in selected soil types were determined (Rosenani et al., 2008). The sludge collected from ten wastewater treatment plants in Malaysia were acidic in nature and the N, P, K, Ca and Mg contents were variable. In general, domestic sewage sludge have lower concentrations of heavy metals compared to the mixed light industry with domestic sludge type. The concentration of trace metal(loid)s in domestic sewage sludge did not exceed the maximum permitted concentrations (MPC) of the European Community Standard (ECS) for land application. Higher N-mineralization rates with or without sludge addition were found in the Bungor and Jawa Series soil. The Serdang series with lower pH and indigeneous organic C had much lower mineralization potentials, even when large amounts of sludge were added. The study highlights the importance of understanding the properties and behaviour of the soil in formulating any sewage sludge-based fertilizer programme for the supply of N for plant growth.

A field trial was also conducted to investigate the potential of sewage sludge as a source of N fertilizer for corn production (Rosazlin et al., 2005) and the uptake of trace metal(loid)s by corn and the accumulation of these metal(loid)s in the soil (Rosazlin et al., 2006). The findings were application of sewage sludge or inorganic N fertilizer $(\text{NH}_4)_2\text{SO}_4$ produced significantly higher yields than the control. The rate of 420 kg N ha⁻¹ sewage sludge gave the highest dry matter yield for the 1st corn cycle and 746 kg N ha⁻¹ for the 3rd corn cycle. It was concluded that the sewage sludge behaves like a slow release fertilizer with only 30% N-mineralized during the duration of corn growth of 70 days duration. Thus, higher rates of sewage sludge is required for fertilization (3 times

N equivalent of the recommended rate of inorganic fertilizer). However, in the 2nd cycle, mineralisation of sewage sludge was slow because of the dry period during this cycle. For sludge to perform efficiently as fertiliser, good moisture conditions are necessary for N mineralisation to take place. The concentrations of trace metal(loid)s in the soil was below the MPC of the ECS and ranged from 12 -35 mg kg⁻¹ Zn, 5-10 mg kg⁻¹ of Cu, 0.6-2.6 mg kg⁻¹ of Cd, 7-24 mg kg⁻¹ of Pb and 7-11 mg kg⁻¹ of Ni. The concentration of trace metal(loid)s in the maize grain ranged from 5-19 mg kg⁻¹ of Zn, 0.6 – 2.6 mg kg⁻¹ of Cu, 0.04 -0.05 mg kg⁻¹ of Cd, 0.03-0.05 mg kg⁻¹ of Pb and 0.7-1.2 mg kg⁻¹ of Ni which were below the MPC values of the Malaysian Food Act 1983 and Food Regulations 1985.

Unfortunately, sewage sludge application is prohibited in myGAP and myOrganic certification. However, sewage sludge is allowed to be used as potting mix for ornamental plants and for urban landscaping a study on the utilization of sewage sludge as peat substitute for potting media with *Chrysanthemum* as test plant was conducted under the project funded by IWK (Plate 1), treatment 2 and 3 display good flowering capability compared to chemical fertilizer (Treatment 1).



Treatment 1, T1: 3soil:2 peat: 1 sand + (Agroblend + foliar)

Treatment 2, T2: 3 soil: 2 (3 ss : 1 cc) : 1 sand + Agroblend

Treatment 3, T3: 3 soil : 2 (3 ss : 1 cc): 1 sand + (1/2 Agroblend + 1/2 foliar)

Plate 1 Plant growth response from sewage sludge potting mix

In another study, Kala et al. 2009 stated that oil palm trunk with sewage sludge at 4:1 ratio was the optimum compost as potting media for ornamental plants because of its suitable texture, not stiff, had high nutrients content (2.05% N, 0.64% P, 1.39% K, 0.71% Ca, 0.23% Mg), pH of 6.2 and low C/N ratio of 19. From the research conducted, the Sewerage Services Department has drawn up the proposed Interim Guideline for Biosolids as fertilizer for non-food crops (Table 5).

Table 5 Guideline for trace metal(loid)s maximum permitted concentration (mg kg⁻¹) for land application of sewage sludge

Trace Metal(loid)s	^aSoil	^bMalaysia	^cEU	^dUSEPA
Cadmium	0.01-7	5	20-40	39
Chromium	5-3000	530	NA	1200
Copper	2-100	800	1000-1750	1500
Lead	2-100	900	750-1200	300
Nickel	NA	200	300-400	420
Zinc	10-300	2000	2500-4000	2800
Mercury	<0.02	8	16-25	17
Arsenic	<0.02	35	NA	41
Molybdenum	<0.02	18	NA	NA

NA: Not Available

^a Typical value for Malaysia Serdang series.

^b Propose Sewerage Service Department Interim Guideline for Biosolids as Fertilizer for Non-Food Crops.

^c European Union Directive (1986) Guideline for Sludge applied with restriction.

^d USEPA Pollutant Concentration Limits for Exceptional Biosolids Applied on Land (Unrestricted sludge application).

Paper Mill Sludge

Paper mill sludge is currently disposed off in the landfill and is costly. The commercial usage of waste paper mill sludge as fertilizer and soil amendment is still at the infancy stage. As the paper mill industries in this country continue to expand, the production of paper mill sludge has also increased. Utilization of organic by-products as soil amendments in agricultural production exemplifies a strategy for converting wastes to resources.

Raw paper mill sludge (RPMS) is massive in nature and less suitable for land application. Thus a bulking agent to increase aeration is required. Since oil palm empty fruit bunch (EFB) can

be easily obtained, it was a good choice to be used as a bulking agent. Raw paper mill sludge were mixed with EFB in 3 ratios, that is 1:1, 1:2 and 1:3 based on volume:volume (Plate 2) (Rosazlin et al., 2011). These compost mixtures had no toxicity effects on plants (Plate 3), had 100% seed germination, high in plant nutrients content, low in C/N ratio and had fine particle size of <18 mm. The concentrations of trace metal(loid)s were also within the recommended level of the Council of European Communities (CEC) for compost. However, it is recommended that compost produced from a ratio of 1:1 is the most suitable for land application.

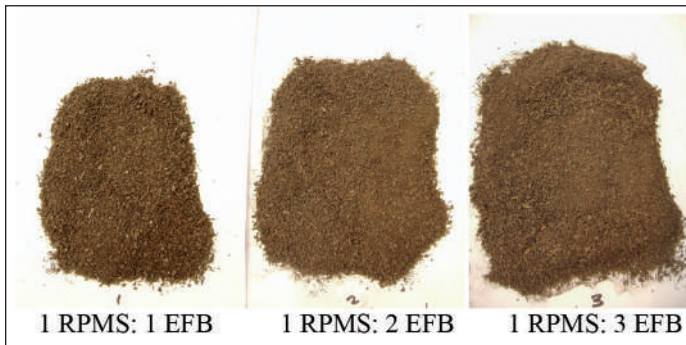


Plate 2 The colour of RPMS and compost mixtures after 90 days of composting

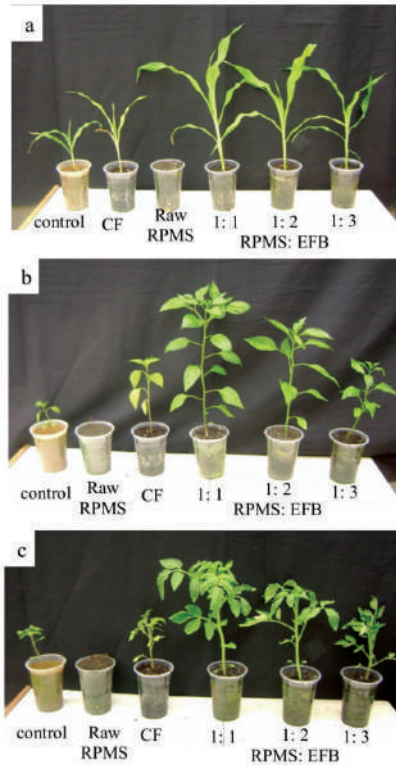


Plate 3 The effect of raw RPM, RPMS and EFB compost mixtures compared to commercial compost on the dry matter weight of a) sweet corn, b) tomato and c) chillies after 14 days planting. The treatment are a) control (mineral soil), b) raw RPMS, c) CF, commercial compost and d) 1:1; 1:2; 1:3 (RPMS:EFB) compost ratio.

A glasshouse study was conducted to evaluate the effect of paper mill sludge on the growth of a forest tree, *Khaya senegalensis* and a medicinal shrub, *Othosiphon stamineus* (misai kucing) (Rosazlin et al., 2015c). Both crops received nutrients input at the early growth after transplanting and growth data was measured at fixed intervals up to a year. *O. stamineus* was measured for above

ground dry matter yield at four crop cycles. Residual nutrients levels in soil after one year of application was also evaluated. Comparison was made with inorganic fertilizer application and untreated soil. *K. senegalensis* responded positively to raw and composted paper mill sludge applications and outperformed the widely used inorganic fertilizer. Paper mill sludge application resulted in higher height increment, diameter growth and total plant biomass than the control and inorganic fertilizer. The medicinal shrub, *O. stamineus*, also produced higher biomass with paper mill sludge application and the trend remains the same for all crop cycles except the relatively lower yield at the 2nd, 3rd and 4th crop cycles. Soil fertility parameters were most affected at the top soil layer with overall accreting tendency under *K. senegalensis* tree with sludge application. Exchangeable potassium was the most limiting nutrient for *K. senegalensis* while P and K were limited for growth of *O. stamineus*.

A field study conducted by Rosazlin et al., (2015b), show that the paper mill sludge has the potential to be a supplementary N fertilizer as well as a soil amendment. The application of recycled paper mill sludge (RPMS) with N significantly contributed to the improvement in plant dry matter (28.5 gram plant⁻¹) and improve soil physical and chemical properties. Total concentration of heavy metal in soil were below the critical values. Hence, the waste paper mill sludge can be successfully used as soil amendment in acidic soil without any serious threat.

Coal Fly Ash

Coal fly ash (CFA) is an amorphous aluminosilicate material, a by-product of coal combustion and is composed of particulate matter collected from flue gas stream. Coal is one of the alternative natural resources used for the production of electricity in Malaysia. The increase use of coal for electric power generation will generate

large quantities of CFA. Kapar power station in Selangor, Malaysia, produced around 200 Mg CFA per day. Currently, only 20% of the CFA is utilized as a component in the cement mixture, the rest is left stacked within the vicinity of the power plant.

Coal is known to contain every naturally occurring elements, and therefore, it is not surprising that CFA can have beneficial effect on solving certain problem of soil quality. Use of CFA as a soil amendment is hindered by the lack of macronutrients (virtually no N and has little plant-available P) in the ash, and also concern on its high concentration of microelements, especially boron (Kukier et al., 2003; Fauziah, et al., 2002). However, boron is a plant nutrient and therefore, CFA can be a source of B fertilizer. The CFA is an alkaline residue produced during the burning of coal for the generation of electricity which is enriched with CaO and MgO and has a pH around 8 to 12. Thus, coal fly ash can supply Ca and Mg to the plants. The pH of CFA can vary depending on the S contents of the coal source, with high S generally producing acidic material and low S producing alkaline material (Fauziah, 1993). The pH of CFA used in this study was 8.34. Nevertheless, CFA is a pozzolanic material or has the capability to act as a cementing material.

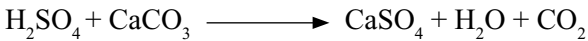
A glasshouse experiment was conducted on two soils of different texture that is Tebuk sandy clay and sandy mine tailings (Fauziah et al. 1999). The soils were treated with different rates of CFA (0, 10, 20, 40, 80 and 160 Mg ha⁻¹) and the uptake of micronutrients (B, Cu, Fe, Mn and Zn) by spinach grown on these soils were determined after 6 weeks of growth. Boron uptake increased while Cu, Fe, Mn and Zn decreased with increasing rates of CFA. The highest spinach dry weight was obtained with the application of 20 Mg ha⁻¹ for Tebuk sandy clay and 40 Mg ha⁻¹ for sandy mine tailings. Thus, CFA cannot be considered as supplementary fertilizer, with the exception of B, and the rates of land-application is limited by B

availability and should be determined prior to its use. However, it has a potential to be a soil amendment because of the slight liming capacity.

Red Gypsum

Red gypsum (RG) is a waste material from the extraction of Ti for industrial purposes. Titanium is extracted from the mineral ilmenite (FeTiO_3) by sulfuric acid digestion. Red gypsum is produced by further increasing the pH of the effluent to about 5.0 by using calcitic limestone (CaCO_3), at which point the remaining sulfate precipitates along with iron oxides. The latter, derived from the iron contents of the ilmenite, are responsible for the red color of the material. Production process of red gypsum can be concluded by using these two equations as shown below.

First stage of reaction:



Second stage of reaction:



Normally, this waste product is disposed off outside the titanium dioxide plant. Such by-product might be suitable for use in agriculture in situations where mined gypsum has been used in the past. Red gypsum can be of great economic value due to its very high Ca and S content. In addition, the presence of the iron oxide responsible for the red color of RG might make it more effective as a soil amendment (Fauziah et al., 1996) rather than as a source of Ca and S fertilizer. Dissolution of the gypsum and subsequent supply of sulphate S to crops might be affected by the presence of the oxides, which have the possibility to adsorb sulphate.

Calcium ions, (Ca^{+2}), is the major exchangeable cation for which gypsum is being evaluated with a view of improving soil physical properties. Particle size (2 mm vs. 212 μm) shows an obvious effect on solubility, with increasing rate of solubility with smaller particle size (Figure 2). For fine-grained red gypsum, differences in solubility kinetics were less pronounced between analytical grade and red gypsum. The importance of including fine-sized particles in surface applied gypsum is that immediate dissolution displayed by these fine particles will maintain a high concentration of electrolytes in the percolating and runoff solutions. The dissolution kinetics of Fe was also monitored, but the concentration is similar to background concentrations of about 1.6 μM or 18 mg/L . This indicates that Fe mainly exist in the non-soluble form.

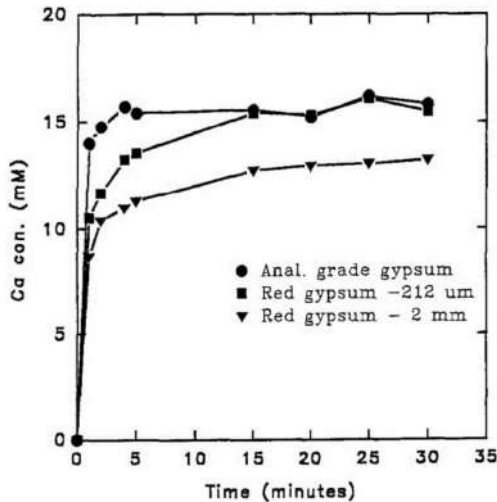


Figure 2 Dissolution kinetic of analytical grade and red gypsum

Red gypsum amendment can be used to temporarily control soil loss from conventional cultivated plots during the growth of the annual crop. In general, gypsum is effective over limited time spans, perhaps the few months needed to establish a crop, but its cost and short life span are major problems for widespread use. With the probable increase in supply of red gypsum produced at the titanium dioxide industry, the economics of gypsum use may improve. Only soils with a high percentage of water dispersible clay gave a positive effect of gypsum or red gypsum on dispersion. (Figure 3)

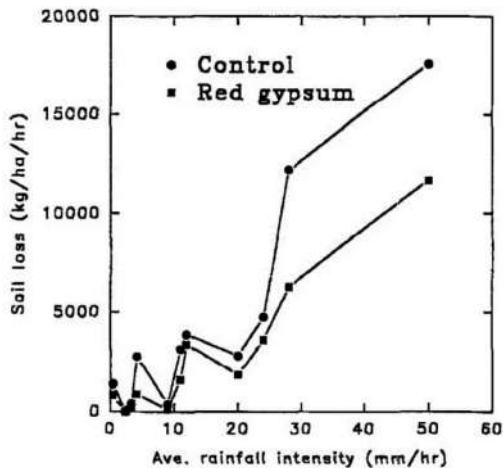


Figure 3 Soil loss versus average rainfall intensity

A glasshouse study was also conducted whereby gypsum was used as a source of Ca for crops requiring additional amount Ca fertilization above the amount supplied by calcitic limestone during the liming process (Chin, 1996). One crop which has high Ca

requirement is the groundnut plant and this Ca is important for the pod-filling of the developed nuts. The result of this study indicated that 2.5 t/ha of red gypsum was required for the Bungor Series Soil and sandy tin tailings to reduce the percentage of empty pods from 15 to 5 % for Bungor Series Soil and 5 to 2% for the sandy tailings.

UTILIZATION OF BY-PRODUCTS FOR *IN-SITU* IMMOBILIZATION TECHNIQUE

Metal(loid)s are not biodegradable and persist for a long time in contaminated soils. It is very expensive and time consuming to remove metal(loid)s from metal(loid)s-rich or contaminated soils. Immobilization of heavy metal(loid)s *in-situ* by adding soil amendment is employed to reduce the bioavailability of metal(loid) and minimize plant uptake. On top of that, some of these amendments can improve soil physical properties such as soil structure and also contain essential plant nutrients and thus improve crop growth.

In-situ immobilization is a cost-effective approach where land-applied amendments are used to stabilize contaminants via adsorption and/or precipitation reactions that render the contaminant immobile (Adriano, 2001). Numerous inorganic amendments such as clays, Al/Fe/Mn oxides and hydroxides may be land applied to metal contaminated soils as means of reducing metal mobility. Nowadays, there is pressure on waste management managers to find ways to convert wastes into resources instead of sending them to the landfill.

Principle Involved in Trace Metal(loid)s Immobilization in the Soil System

The basic principle involved in the immobilization technique, the metal(loid) of concern is removed from soil solution either

through adsorption, complexation, and precipitation reactions, thereby rendering the metal(loid) unavailable for human and plant uptake and leaching to groundwater. Heavy metal incorporation in the soil is controlled by adsorption processes, such as surface complexation and ionic exchange, but other mechanisms such as precipitation are likely to contribute to metal retention in the soil (Sastre et al., 2006). Adsorption is defined as the accumulation of ions at the interface between a solid phase and an aqueous phase. Adsorption isotherms have been widely used in studies on adsorption phenomena, supplying numerical parameters that provide information on the retention capacity and intensity of the metal by the soil. The advantage of these equations is that they can be applied to adsorption of any ions and gives straight forward parameters which can be related to soil properties.

Another meaningful study to complement the adsorption isotherm study is the widely-used sequential extraction or fractionation study. This technique is used for understanding element distribution in the solid phase. These methods are based on the rational use of a series of more or less selective reagents chosen to solubilise successively the different mineralogical fractions thought to be responsible for retaining the larger part of the metal(loid)s. They are intended to simulate the various possible natural and anthropogenic modifications of environmental conditions.

Sorption

One of the important criteria for effective adsorption of metal(loid)s to by-products (sorbent) is the particle size of the by-products. The finer the size of the by-products, the higher will be the surface area, hence the higher is the adsorption of the metal(loid)s. However,

there is a limit to the fineness of the particle size and should be greater than 100 μm . Retention of charged metal(loid) solute species by charged surfaces of soil components is broadly grouped into specific and non-specific retention. Non-specific adsorption is a process in which the charge on the ions balances the charge on soil particles through electrostatic attraction whereas specific adsorption involves chemical bond formation between the ions in the solution and those in the soil surface. In other words, specific adsorption involves the exchange of metal(loid)s cations with surface ligands of the soil colloids to form covalent bond. The metal(loid)s most able to form hydroxyl complexes are specifically adsorbed to the greatest extent. Thus, in specific adsorption, the metal(loid)s are held tighter or closer than the non-specific adsorption and are more difficult to come out into the solution. Generally, the trace metal(loid)s are specifically adsorbed to the soil colloidal surfaces.

Precipitation

Precipitation appears to be the predominant process in high pH soils in the presence of anions such as SO_4^{-2} , CO_3^{-2} , OH^- and HPO_4^{-2} , and when the concentrations of the metal(loid) ion is high. Precipitation of metal(loid) phosphates/carbonates is considered to be one of the mechanisms for the immobilization of metal(loid)s such as Cu and Pb, especially in substrates containing high concentration of metal(loid)s. Similarly, liming typically enhance the retention of metal(loid)s. In general, metal(loid)s tend to be soluble at low soil pHs and precipitate out at higher pHs.

Co-precipitation of metal(loid)s is another mechanism of metal(loid)s immobilization in the soils system. Co-precipitation is the simultaneous precipitation of chemical agent in conjunction with other elements by any mechanism at any rate. Metal(loid)s

are precipitated with clay minerals, hydrous Fe and Mn oxides and calcite (Table 6). Arsenate (As (V)) sorption onto ferrihydrite showed that co-precipitation was a more efficient process than sorption for metal(loid) removal from aqueous solutions. Liming the soil can bring out Cd of the soil solution as it will co-precipitate with CaCO_3 .

Table 6 Co-precipitation of trace metal(loid)s with soil minerals

Mineral	Co-ppt trace metals
Fe oxides	V, Mn, Ni, Cu, Zn, Mo
Mn oxides	Fe, Co, Ni, Zn, Pb
Ca carbonates	V, Mn, Fe, Co, Cd
Clay Minerals	V, Ni, Cu, Cr, Zn, Cu Pb, Ti, Mn, Fe

(Alloway, 1995)

Oxidation

Trace metal(loid)s, including As, Cr and Hg are most commonly subjected to microbial oxidation/ reduction reactions, thereby influencing their speciation and mobility. Such metal(loid)s can exist in various forms or species with several valences based on the soil condition, whether it is well drained (oxidized) or waterlogged condition (reduced). Example, As can exist as As (V) under oxidized condition and become immobilized, and As (III) under reducing condition and become mobile.

Under reducing condition also, sulphate are reduced to sulphide at $p_e = -2.0$, which can lead to precipitation of metal sulphides of elements such as Cd, Co, Cu, Ni, Pb and Sn. The sulphides of these elements are quite insoluble, so their mobility and phytoavailability

are considerably less than would be expected under well-oxidized soils. Exception are Fe and Mn, in that they are more soluble under reducing than in oxidizing conditions.

Methylation

Methylation is a biological mechanism for the removal of toxic trace metal(loid)s by converting them to methyl derivatives that are subsequently removed by volatilization. Typical examples of such metal(loid)s are As and Hg. Although methylation of metal(loid)s occurs through both chemical (abiotic) and biological processes, biological methylation (biomethylation) is considered to be the dominant process in soils and aquatic environments.

Agricultural and Industrial By-products Selected for *In-situ* Immobilization of Trace Metal(loid)s

Studies have been conducted on selected amendments for the immobilization of trace metal(loid)s (Table 7). These amendments can be categorized as liming materials, organic materials or biosolids, phosphate compounds and metal oxides (Fe-rich materials).

Table 7 Selected soil amendments in the immobilization of metal(loid)s in soils

Amendments	Metal(loid)s	Observations	References
Liming materials			
Ground magnesium limestone	Mn	Manganese concentration in the soil solutions decreased exponentially with increasing pH after application of ground magnesium limestone.	Shamshuddin et al., 2009
Red gypsum (RG)		RG and WTR can be recommended as soil amendments because of their effectiveness in reducing metal(loid)s in the soil through changes in soil pH. However for WTR, P deficiency in plant has to be monitored because of WTR ability to fix P.	Fauziah et al. (2011), Nur Hanani et al. (2008)
Water treatment residue (WTR)	Cd, Cu, Ni, Pb and Zn		

<p>Organic materials/ Biosolids Biochar – Oil palm empty fruit bunch (EFB) and rice husks (RH)</p>	<p>Cd and As</p>	<p>Oil palm EFB and RH biochars indicate that the commercially produced biochars have good potential to be used as sorbents for As and Cd.</p>	<p>Sari et al., 2014</p>
<p>Coal Fly ash</p>	<p>Cd, Cu, Ni, Pb and Zn</p>	<p>CFA can significantly reduce the Zn derived from sewage sludge from being taken up by the maize plants at a ratio of (10%:5%) and up to ratio 4:1 (20%:5%) coal fly ash to sewage sludge. However, CFA was not a good liming agent since the calcium carbonate equivalent (CCE) was low.</p>	<p>Nur Hanani et al., 2010</p>

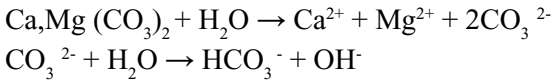
Phosphate compounds

<p>Egyptian rock phosphate (ERP) Bone meal (BM) Triple super phosphate (TSP)</p>	<p>Pb</p>	<p>Applications of P-amendments show effectiveness of TSP in reducing lead in soils. The best treatments are as follows; TSP >ERP >BM. These treatments were recorded able to stabilize the Pb as indicated in the percentage reduction in phytoavailable form pools into a more stable form of complex.</p>	<p>Naim et al. (2017)</p>
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Liming Materials

Malaysian soils dominantly fall (about 75%) into the Ultisol and Oxisol Orders in Soil Taxonomy. These soils are generally acidic, pH of 4.0-5.0 and contain essentially of variable charge minerals, namely sesquioxides and kaolinite, thus, have low cation exchange capacity or cations retention capacity. In Malaysia, liming is the most common management practice used to overcome the problems associated with soil acidification. Most plants grow well at a pH range of 5.5–6.5 and liming is aimed to maintain the pH at this range. The main purpose of liming is to reduce aluminium toxicity in highly weathered acidic tropical soil, but at the same time, this practice can help reduce heavy metals availability to plants via precipitation process. Liming experiments on typical Ultisols and Oxisols have indicated the need for liming for annual crop production. In a study on an Oxisol grown with cocoa, the application of lime at 2 t ha^{-1} reduced soil solution Mn concentration in the 0-15 cm layer from 27 to $12 \mu\text{M}$ after 3 months (Shamshuddin et al., 1991). Manganese concentration in the soil solutions decreased exponentially with increasing pH after application of ground magnesium limestone (Figure 4). Applying lime in combination with gypsum would bring more Ca and/or Mg further down the soil profile, thus alleviating to some extent subsoil acidity. Applications of ground magnesium limestone (GML), usually known as dolomitic limestone, would also supply the necessary Ca and Mg needed for corn and groundnut growth. The presence of more Ca in the soils arising from lime and/or gypsum applications is also beneficial because Ca can to a certain extent alleviate Al toxicity. The increase in solution pH would certainly affect the availability of other metal(loid)s in the soil.

The increase in solution pH resulting from GML application is due to production of hydroxyl ions when GML is dissolved and subsequently hydrolysed:



The hydroxyl ions then reacts with Al in the solution to precipitate as aluminum hydroxide, which over time may crystallize into gibbsite $[\text{Al}(\text{OH})_3]$:

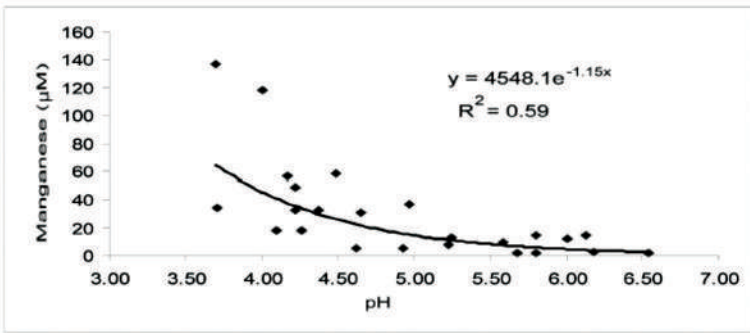
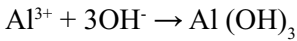


Figure 4 Relationship between soil solution Mn and pH (Shamshuddin et al., 1991)

Biochar

Biochar is a carbonaceous material which can adsorb metal(loid)s in soils and water. The main factors influencing the sorption behaviour of biochars are pyrolysis condition and the feedstock type in the production of biochars. One type of biochar may not be appropriate for all cases of remediation. The application of biochar to soil may improve the sorption capacity of metal(loid)s in soil. This carbonaceous product was reported to have many functional groups with high surface areas, which are likely related to its potential to act as an adsorbent.

In terms of remediation of heavy metal contaminated soils through their retention in the soil system, biochar has been considered to be potentially effective. A study by Beesley and Marmiroli, (2011) have demonstrated that biochar has a high capacity to adsorb pollutants in contaminated soils. Biochar can stabilize the heavy metals in the contaminated soils, improve the quality of the contaminated soil and has a significant reduction in crop uptake of heavy metals.

Biochar is a fine-grained charcoal-like material produced through pyrolysis, which is heating of biomass to temperature of 300-600 °C under air deprived conditions. Through pyrolysis, the feedstock changes chemically to form structures that are more resistant to microbial degradation than the original material.

Utilization of biochar as a soil amendment has attracted great interest globally due to the apparent benefits to soil fertility and plant growth as well as the potential to store or sequester C in the soil system. It has been reported that activated carbon, which is a subset of biochar, had been used as a substrate to improve the adsorption of heavy metals such as mercury, a process which is termed as chemisorption. The mechanism of heavy metal retention in soil by biochar can be categorized as physical or chemical in nature. The physical aspects deal more with filtering mechanism of the heavy metal due to its structure or size by the pore size of the biochar. It is important to characterize the pore size distribution of biochar, the percentage of macropore, mesopore, nanopore, because the type of pores dictate the extent of liquid-solid adsorption processes.

In Malaysia, a pilot scale biochar manufacturing plant using a modern engineering system has been built by Universiti Putra Malaysia (UPM), in collaboration with a private company (Nasmech Technology Sdn. Bhd.). The plant was built to produce biochar from oil palm empty fruit bunches (EFB) and is capable of producing

20 t of biochar daily. Additionally, biochar derived from rice husks has been produced commercially in Malaysia to avert wastage of large quantities of rice husks (RH). It is reported that 97,980 million tonnes of rice husk was produced annually during the processing in the mills (Bernas Sdn. Bhd.).

The surface morphology of biochar samples was observed under Jeol JSM-6400 scanning electron microscope (Sari et al., 2014). Plate 4 shows that EFB biochar possesses uniform pores and smooth wall surfaces with maximal 20 μm in diameter. Small particle-like ashes were found scattered on the surface area of EFB biochar as observed in Plate 4. In comparison, the pores on rice husk biochar are not well-shaped with diminished structure of pores (Plate 5). Small pores on the rough rice husk biochar surface was observed as shown in Plate 5. Pyrolysis temperature can attribute to the pores formation and destruction on biochar. When low temperature was applied, the biochar cell structure and arrangement was found similar to the cell structure and arrangement of the original biomass (Pavithra, 2011). The stack of biochar cells and pores were arranged accordingly and well-shaped as found in the SEM image of EFB biochar. However, as the temperature increase, the pore size become enlarged and the walls between adjacent pores were destroyed (Zhang et al., 2004), which explained the diminish pores on rice husk biochar. The lack of biochar structure also might be due to the volatilization process during the biochar production.

From the observation on the SEM images (Sari et al., 2014), both biochar generally exhibit macropores with internal diameter size of 10 μm . The macroporosity (>50 nm) of biochars are relevant for soil aeration and water movement (Troeh and Thompson, 2005). Macropores also facilitate the root movement through the soil and act as habitats for the soil microbes (Saito and Muramoto, 2002). Hence, biochar has the potential to improve soil physical properties

such as soil water retention and porosity. Basso et al. (2013) reported the addition of biochar on sandy loam soil increased the water-holding capacity by 23% compared to the non-amended soil. The soil moisture at field capacity also increase with the increase of char surface area and porous structure. The macropores are also important as feeder pores to transport adsorbate molecules to the meso- and micropores.

A mixture of meso- and micropores were also present on EFB and rice husk biochar surface. The micropores of biochar make the greatest contribution to total surface area, hence responsible for the high adsorption capacities of molecules (Rouquerol et al., 1999). Mesopores are also of importance for many liquid-solid adsorption processes, as reported by Lua et al., (2004), on pistachio-nut shells. Thus, based on the EFB and rice husk biochar structural surface, they have the potential to sorb metal and metalloid to reduce the mobility of these trace elements in soil.

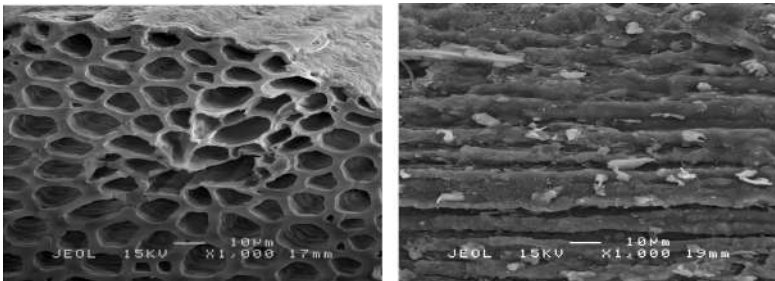


Plate 4 SEM image of EFB biochar at 1000 x magnification (Sari et al., 2014)

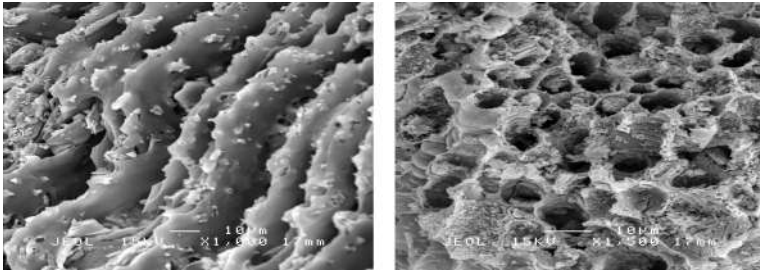


Plate 5 SEM image of rice husk biochar at (a) 1500 and (b) 1000 x magnification (Sari et al., 2014)

The Brunauer, Emmett and teller (BET) surface area of biochar indicates the physical changes of biomass during the pyrolysis process. The surface area depends largely upon the carbon (C) mass removed during the processing, creating pores in the materials (Zabaniotou et al., 2008). The sorption ability of biochar can be determined from its surface area, where high surface area will increase the sorption capacity. Surface area and porosity of EFB and rice husk biochar are presented in Table 8.

Table 8 BET surface area and porosity of biochars

Biochar	BET surface area (m ² /g)	Pore volume (cm ³ /g)	Pore surface area (m ² /g)	Average pore diameter (nm)
EFB	46.32	0.01	0.61	3.85
Rice husk	23.22	0.01	1.41	4.34

Biochar produced from EFB had a larger surface area than RH biochar. The higher surfaces area of EFB biochar may indicate the adsorption capacity of heavy metals compared to RH biochar.

In general, biochar surface areas can be influenced by biochar's micropore volume, choice of feedstock and pyrolysis processing condition (Boateng et al., 2007). The micropore volume of EFB biochar was found to be the same as RH biochar ($\sim 0.01 \text{ cm}^3/\text{g}$). Internal surface area of biochar which represent pore on the inner wall resulted from interior crack was referred to as micropore area. Meanwhile, the average pore diameter for both biochar are in the range of mesopores diameters, with the internal pore width between 2 to 50 nm. This indicates the potential of adsorption capacity EFB biochar and RH biochar in liquid-solid adsorption (Lua et al., 2004).

The adsorption isotherm data were fitted to the Langmuir's adsorption model. Table 9 shows the values of adsorption isotherm parameters for EFB biochar and RH biochar. The maximum adsorption capacity (q_{max}) of EFB biochar for As was 0.424 mg g^{-1} , which is higher than RH biochar (0.352 mg g^{-1}). Similar trend was found on adsorption maximum, q_{max} , of Cd with the values of 15.15 and 3.19 mg g^{-1} , for EFB biochar and RH biochar, respectively. The parameter b (binding affinity) is related to the affinity of the binding sites, which allows comparisons of the affinity of biochar toward the metal(loid) ions. EFB biochar had a higher affinity for As than did RH biochar. In contrast, the binding affinity (b) of Cd for RH biochar is higher than EFB biochar. There are several factors attributed to sorption mechanism of trace elements with addition of biochar, of which the most important are pH and CEC. The alkaline properties of biochars increased the solution pH, which induced metal immobilization through metal precipitation and decreases metal solubility. Value of R^2 shows correlation or linear relationship, whereas the relationship become more linear when the value is closer to 1. The high correlation coefficient values (R^2) which ranged from 0.98 to 0.99 indicate that the Langmuir isotherm best fitted the experimental data.

Table 9 Sorption isotherm obtained by fitting the data with the Langmuir isotherms for the EFB biochar and RH biochar

Biochar	Heavy metal	Langmuir model		
		q_{\max} (mg g^{-1})	b (L mg^{-1})	R^2
EFB	As	0.4240	0.7299	0.9890
	Cd	15.1515	0.1142	0.9921
RH	As	0.3522	0.0248	0.9823
	Cd	3.1908	0.6920	0.9984

Samsuri et al. (2013) reported coating the biochars with Fe (III) greatly increased their adsorption capacities for both As (III) and As (V). The results indicate that the commercially produced EFB and RH from Malaysia have good potentials to be used as adsorbents for As (III) from aqueous solutions. Furthermore, coating the EFB and RH with Fe (III) increased their adsorption capacities for both As (III) and As (V) making the biochars more effective as adsorbents for both As (III) and As (V).

Soil solution study of incubated arsenic-rich Histosol amended with biochar was conducted to evaluate the effects of EFB biochar and RH biochar on water-soluble As naturally present in Histosol (Figure 5) (Sari et al., 2014). Empty fruit bunch and RH biochars exhibited important feature as adsorbent with the porous structure and alkaline properties. The sorption experiment has shown the potential of these biochars to immobilize As in the soil system (Sari et al., 2014). Other studies conducted on the ability of biochars to adsorb As have shown negative results. It is however believed that the Fe present (1.0 %) in the Histosol has coated the surfaces of biochar particles applied to the soil, and these coatings have adsorbed or immobilized As in the soil system. Thus, both

the decreased of As concentration and increased of soil pH in soil solution study indicate the ability of biochar to reduce the phytoavailable As in contaminated soil (Figure 6).

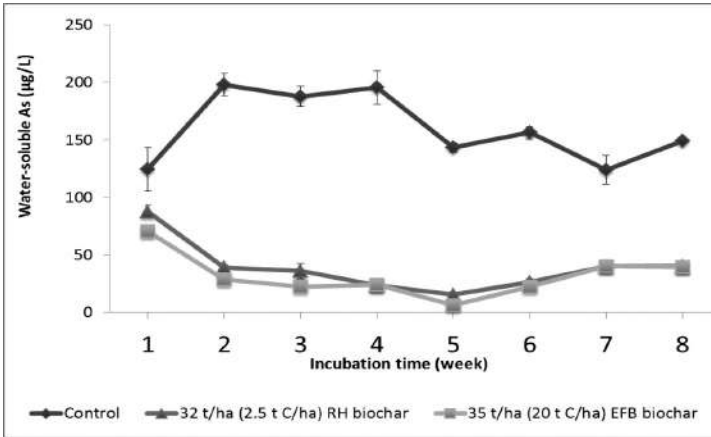


Figure 5 Effects of EFB and RH biochars on water-soluble arsenic in pore water

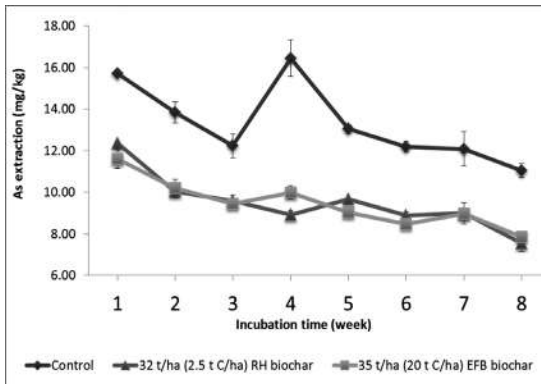


Figure 6 Effects of EFB and RH biochars on extractable arsenic in soil

The pot experiment was conducted to determine the optimum rates of biochars to reduce arsenic (As) uptake by sweet corn (Figure 7). Two types of biochars, EFB and RH with 5 rates (0, 2.5, 5, 10, 20 t C/ha) application were applied to 15 kg naturally contaminated soil in polybag. After 56 days of growth, biochar reduced the concentration of As in foliar tissue by 58% and 61% with the highest application of EFB and RH biochars, respectively, at 20 t/ha C compared to the non-amended soil. This further proved that As can be adsorbed by the Fe coating present on the surface of biochars and hence prevent it from being taken up by the corn plants. Thus, this study further complement the fact that the biochars are effective in reducing the availability of As taken up by sweet corn as its concentrations decreased with increasing rate of biochars.

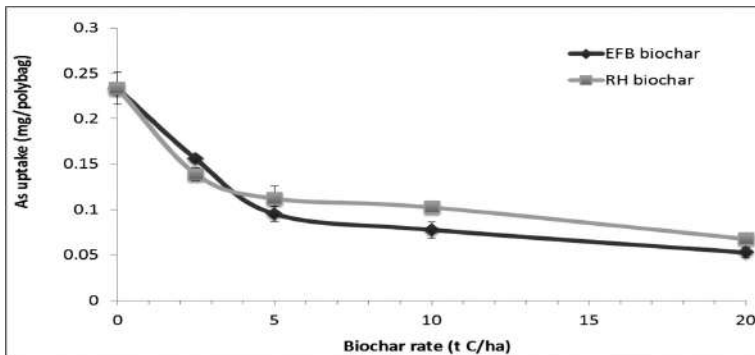


Figure 7 The uptake of arsenic by sweet corn plant after 56 days of planting

Table 10 Industrial by-products amendments to sewage sludge (5%) treated soils (glasshouse study)

Amendments	Metal	Effects	Reference
Red Gypsum	Cu	Decreased sig. at > 10% level	Fauziah et al., 2011
	Zn	Decreased significantly at > 5% level	
	Fe	Decreased sig. at 20% level	
	Cr	Decreased sig. at 2.5 to 10% level. At 20% highest Cr level – not sig. different from control	
Coal Fly Ash (CFA)	Cu	Decreased significantly at 5% - 10%, but increased sig. at 20%	Nur Hanani et al., 2010
	Zn	Decreased significantly at greater than 5% treatment	
	B	Increased significantly compared to control	
Water treatment residue (WTR)	Cu	Decreased significantly compared to control	Nur Hanani et al., 2008
	Zn	Decreased significantly compared to control	

Industrial By-products/Minerals

Addition of industrial by-products to soil has gained importance recently as an alternative to remediate trace metal(loid)s contaminated soil (Table 10). For example, by-products from steel (iron oxides) and energy industries (ashes) will be assessed as an environmentally and resource-efficient option due to their alkalinity or acid neutralizing capacity and high specific surface area. The ANC is usually expressed as CaCO_3 equivalent and one of the most important factors used to evaluate the value of industrial by-products to be used as a liming agent on acidic soil.

Red Gypsum

The red gypsum studied was alkaline in nature, with a pH of 7.98 due to the presence of residual CaCO_3 in RG (Fauziah et al., 1996). This property can be exploited to reduce the solubility and hence, phyto-availability, of some trace metal(loid)s in the soil system. The acid-neutralizing capacity is the most important characteristic in the evaluation of the value of the material as a liming agent. Red gypsum is not a good liming agent, with only 1.79% calcium carbonate equivalence. However, high rates of application ($> 2.5\%$) can have significant influence on the pH of the soil system (Nur Hanani et al., 2009). The surface area for the red gypsum (pulverized and sieved through 2.0 mm sieve size) was $39.8 \text{ m}^2 \text{ g}^{-1}$. The high surface area plays a central role for adsorption behaviour. Furthermore, the presence of Fe oxide can contribute to the co-existence of positive and negative charges on the variable charge oxide surface (Plate 6).

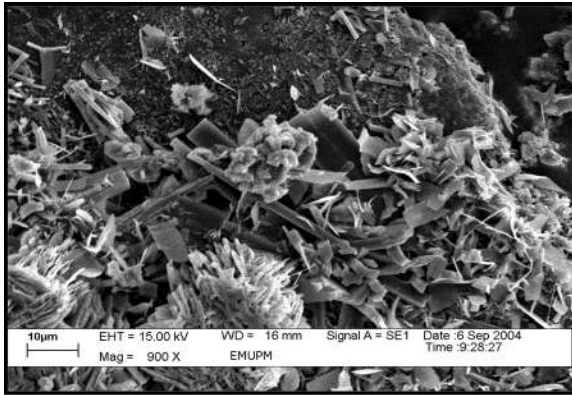


Plate 6 Fibrous crystal aggregates of gypsum with some coatings of iron oxides

A column leaching study was conducted to investigate the potential utilization of red gypsum for *in-situ* immobilization of arsenic in the soil system. In this experiment, the treatment used was the different rates of red gypsum. The treatments were applied at the top of the soil only. The treatments were: T1: no red gypsum (control), T2: 25 t/ha red gypsum, T3: 50 t/ha red gypsum, T4: 100 t/ha red gypsum. From this study, red gypsum application has the potential to immobilize arsenic in the soil system and thus prevent arsenic from being taken up by the crop grown on arsenic contaminated soil. The presence of Fe in red gypsum can help surface adsorbed or co-precipitate As in the soil system (Figure 8 and 9).

Solid Waste Management

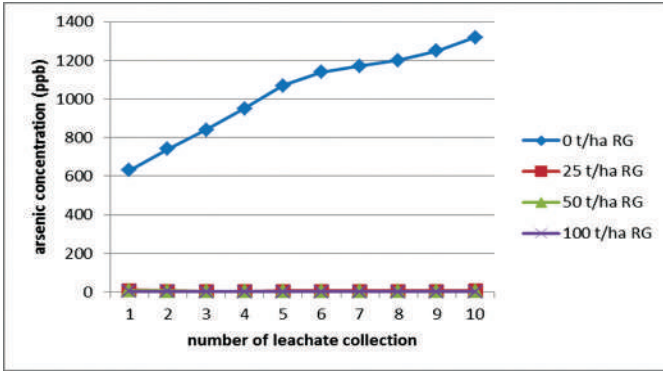


Figure 8 Arsenic concentrations in each leachate collection (50 mL) up to one pore volume for each treatment

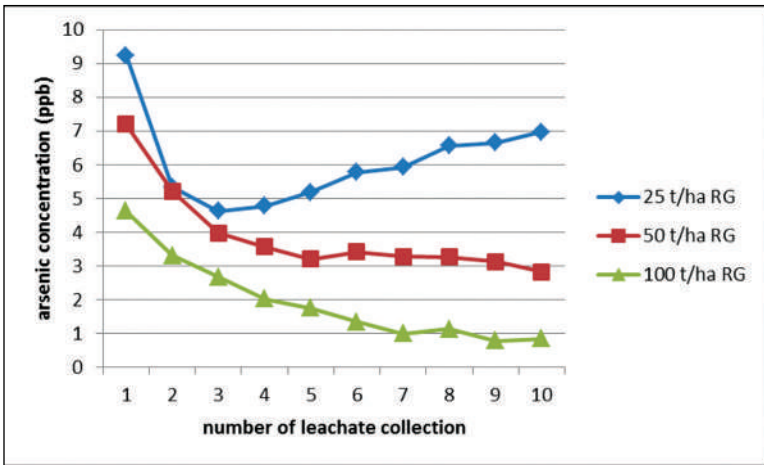


Figure 9 Arsenic concentrations in each leachate collection (50 mL) for 25 t/ha, 50 t/ha and 100 t/ha red gypsum

In a soil incubation study, RG was applied to sewage sludge treated soil (Fauziah et al, 2011). Sewage sludge tend to have high concentrations of Cu and Zn and its application to soil increase these metals content. There seemed to be a lag-phase in the release of Zn from the RG minerals into the soil solution (Figure 10). The Zn concentrations in the soil solution started to increase only after five weeks of incubation for the treatments with low rates of RG application. The reason for this slow release of Zn into the soil solution is not known. However, this study demonstrated that increasing the RG amendment rates (5%, 10%, 20% and 40%) clearly reduced the Zn concentrations in soil solution after ten weeks of incubation. Thus, RG has the potential to fix Zn in the soil system and make it less phytoavailable.

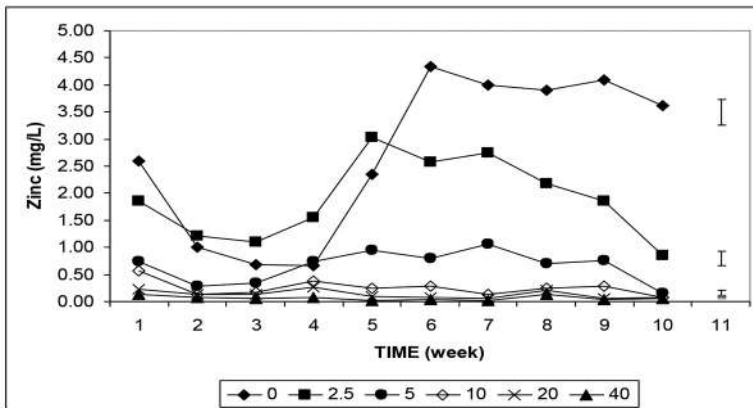
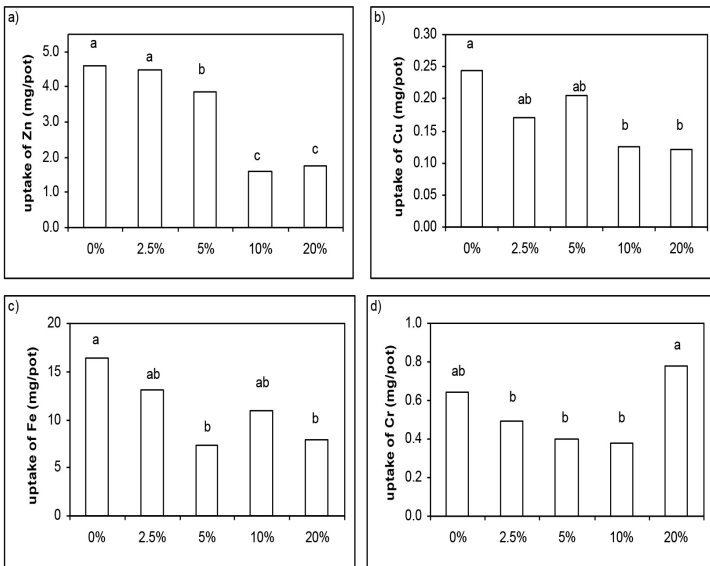


Figure 10 Soluble Zn at different rates of RG treatments of contaminated soil

Increasing the rate of red gypsum application resulted in decreasing uptake of Zn, Cu and Fe by the corn plants (Figure 11). This is due to the increase in soil pH. The residual alkalinity plus the buffer capacity of iron oxides (goethite and hematite) (Fauziah

et al., 1996), allow red gypsum to consume protons from an acid soil. However, the results for Cr seemed to be rather varied. The levels of Cr in red gypsum were found to be low. This study did not ascertain whether the organic matter in sludge alleviates the effect of excess Ca and Fe in the mixed soil system. This needs to be investigated. Furthermore, co-mixing two products such as RG and compost can turn the by-products into a more useful soil amendment as the amending capability of the by-product can be complemented and further enhanced by the co-mixed by-products (Fauziah et al., 2011).



Similar letters above bars indicate that the values represented by the bars are not significantly different at the 1% level, according to the Duncan New Multiple Range Test (DMRT)

Figure 11 Uptake of microelements (mg/pot) using contaminated soil amended with red gypsum

A glasshouse study was then conducted with the same treatments as the soil incubation study using sweet corn as the test crop. Two set of experiments were established with 4 treatments and 4 replicates. Treatments of experiment are: Red Gypsum + EFB Compost with different rate of red gypsum (2.5, 50, 100 and 200 t/ha) (Table 11). For Fe concentrations, application of RG+EFB compost show significant decrease in Fe concentrations in foliar tissues at the rate of 100 and 200 t/ha compared to the lower rates. The toxic level of Fe for corn is >350 mg/kg, thus there is no problem of Fe toxicity to plants in this case. For Zn concentration, significant decrease in Zn concentration were found at the rates of 100 and 200 t/ha compared to the lowest rate of RG. For Cd, significant decreased in Cd concentrations with the increasing rates of RG+EFB compost were found at the rates of 100 t/ha and 200 t/ha compared to the lowest rate of RG+EFB compost used.

Table 11 Effects of treatments on trace metal(loid)s in foliar tissues

Treatment	Fe	Zn	Cd	Cr
	mg/kg			
RG 2.5t/ha + EFB Compost	305 ab	83.75 a	0.16 ab	0.98 ab
RG 50t/ha + EFB Compost	477 ab	75.65 a	0.08 ab	0.07 b
RG 100t/ha + EFB Compost	237 bc	39.20 b	0.03 b	0.14 ab
RG 200t/ha + EFB Compost	94 bc	9.63 b	0.05 b	0.06 b

Means having the same letters within column are not significantly different at $p > 0.05$

Co-application of red gypsum amended soil with biosolids was carried out in field condition with five treatments and four replicates at Lanchang, Pahang. Sweet corn (*Zea mays* L.) was used as the test crop. Rate of red gypsum used was 100 tonnes/ha combination with EFB compost, EFB biochar and chicken dung in the ratio of

1:4 volume/volume basis. Based on (Table 12), co- applications of RG with biosolids shows a decrease in Cd concentration compared to the control. The Cd concentration is above the 95th percentile; nevertheless, it is below the maximum allowable limit of the Canadian soil regulation. For Cr and Ni concentrations, application of RG: EFB compost shows a significant decrease in Cr and Ni concentrations compared to the control. For Pb concentrations, application of RG: EFB compost shows significant decrease in Pb concentrations compared to the control and other treatments. Application of RG and biochar shows significantly higher Pb concentration compared to the control and other treatments. Chromium, Ni and Pb are below the 95th percentile or investigation level and also below the maximum allowable limit of the Canadian soil regulation. In conclusion, application of RG and EFB compost shows the best co-application on soil towards the corn growth based on the significant effect on the uptake of trace metal(loid)s.

Table 12 Co-application of red gypsum amended soil with biosolids (field study)

Treatment	Cd	Cr	Ni	Pb
	mg/kg			
Control	3.81 a	1.93a	0.22a	3.49a
RG only	3.79 ab	1.59ab	0.19a	3.79ab
RG: EFB Compost	2.74 c	0.21c	0.05b	0.62d
RG: Biochar	3.20b	1.35ab	0.26a	3.92a
RG: Chicken Dung	3.17b	1.44ab	0.22a	2.73c
95 th percentile- Investigation Level	0.30	60	45	65
Maximum Allowable Limit (Canada)	8	75	100	200

Means having the same letters within column are not significantly different at $p > 0.05$

Coal Fly Ash

Alkaline agent can be used as a stabilization agent for contaminated soil to reduce pathogen and trace metal(loid)s availability. Large surface area determination of CFA was probably due to large number of spongy irregular carbon-rich particles of unburnt coal (Fauziah, 1993). Surface area determination for this CFA was $7.5 \text{ m}^2/\text{g}$. Hence, the particle size distribution will provide information relating to land application of the ash, in term of trace elements solubility and effect on soil physical properties (Plate 7).

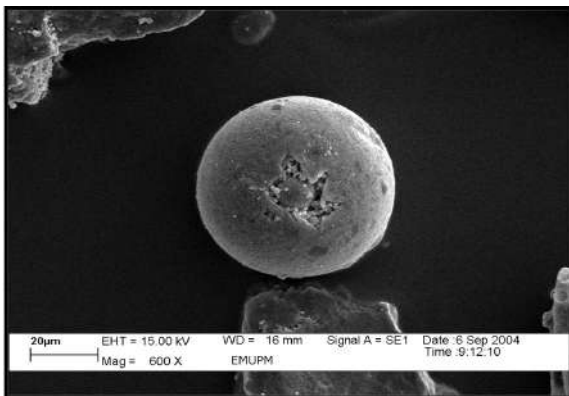


Plate 7 Cenosphere shape of Coal Fly Ash

The neutralization of acid by CFA is a relatively slow process that mainly involves the particle surfaces (Wong et al., 2002). The CFA was not a good liming agent, with only 0.504% CaCO_3 equivalent (CCE). Based on the low level of Ca in this CFA, it is considered only as a Class F fly ash (Bilski et al., 1995). Therefore, considerably large quantities of this CFA compared to lime will be required to raise the pH of soil to some target level.

A soil incubation study was also conducted whereby, CFA was applied on sewage sludge treated soil (Nur Hanani et al., 2010). Increasing the CFA amendment rates clearly reduced the Zn concentrations in soil solution for the ten weeks of incubation (Figure 12). The reduced concentration of Zn probably can be explained by the higher adsorption and precipitation of Zn with an increase in pH. The control treatment (0% CFA) had Zn concentrations in the soil solution ranging from 1.47 mg L⁻¹ to 0.67 mg L⁻¹ for the four weeks of incubation and increased drastically at week five to 5.0 mg L⁻¹. It is not known with certainty why there was a delayed dissolution of Zn from the sewage sludge. The Zn concentration for the control treatment after week five until week ten of the incubation was still high (> 3.73 mg L⁻¹) compared to other treatments. Treatments using 2.5% and 5% CFA ranged less than 2.15 mg L⁻¹ whereas treatments using greater than 10% CFA had the lowest Zn concentrations which were less than 1 mg L⁻¹. This indicates that CFA was feasible as a stabilization agent to reduce trace metal(loid)s toxicity in the sewage sludge-treated soil.

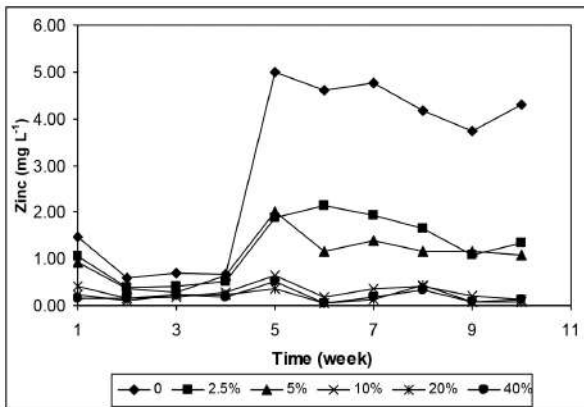
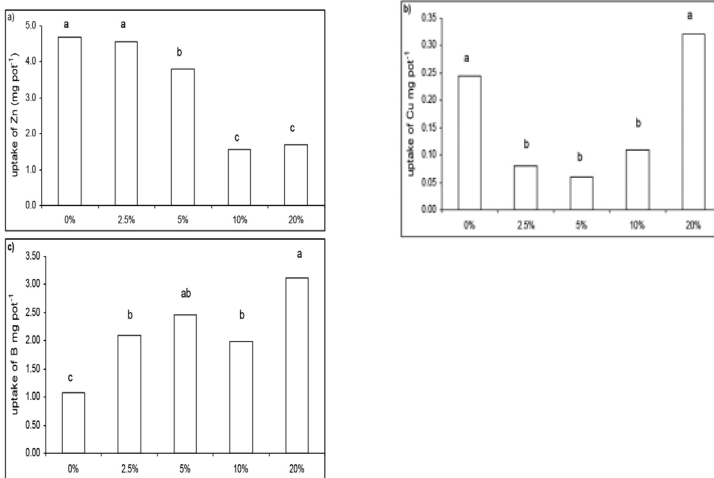


Figure 12 Soluble Zn at different rates of CFA treatments of contaminated soil

The Zn uptake by maize for treatment using CFA is shown in Figure 13. Overall, the concentration of Zn uptake by maize significantly decreased at higher rates of CFA treatments. Usage of 2.5% CFA did not show any significant result as compared to the control treatment. However, addition of more than 5% CFA significantly reduced Zn concentration in maize. This showed that the CFA was useful as a soil amendment to fix Zn in the contaminated soil.

The results showed application of CFA up to 10% reduced Cu uptake by the maize plants compared to the control (Figure 18). However, there was no significant difference in Cu uptake by maize between the control and the 20% CFA treatment indicating that CFA can be beneficial as a soil amendment to reduce Cu uptake by plant but, the amount of CFA should be applied at a proper rate to avoid Cu toxicity (Nur Hanani et al., 2010).



Note: Similar letters above the bars indicate that they are not significantly different at 99% confidence level, according to the Duncan New Multiple Range Test (DMRT)

Figure 13 Uptake of trace metal(loid)s (mg/pot) using soil amended with CFA

Drinking-water Treatment Residues

In Malaysia, a low-cost and potentially effective substitute for remediation could be drinking-water treatment residues (WTRs). The pH for WTR for this study was close to being neutral. The WTR has a pH of 7.07, the mineral present in WTR, such as kaolinite, gibbsite and Fe-oxides, provide surfaces for the adsorption of heavy metals. Value for WTR surface area was $28.3 \text{ m}^2/\text{g}$ and this value was largely dependent on the size of the sample which was less than 2 mm due to the grinding process. Surface area determination can be used to estimate the amount of surface sites available for surface complexation reaction (Plate 8). Butkus (1998) reported a surface area of WTR of $10 \text{ m}^2/\text{g}$. Dzombak and Morel (1990) estimated that WTR can bind with protons, cations and anions based on the range of sorption maxima reported from $160 \text{ m}^2/\text{g}$ to $600 \text{ m}^2/\text{g}$. The ANC of WTR was 0.504% CCE. Thus, WTR cannot be considered a good liming material compared to the pure CaCO_3 , but perhaps usage at high rates of this material can still increase the pH of acidic soil.

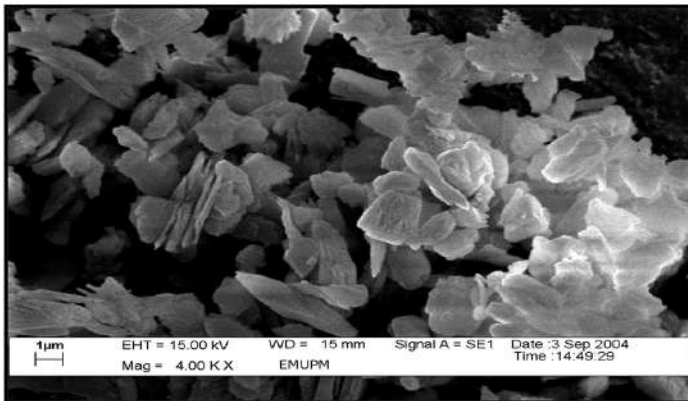


Plate 8 Presence of kaolinite (hexagonal shape) and illite flakes of WTR

Also, in an incubation study of sewage sludge treated soil, treatment using the highest rates of WTR (40%) gave the lowest Zn concentration in the soil solution (Figure 14) (Nur Hanani et al., 2008). Perhaps, the high Zn concentrations and high pHs at the higher WTR rates led to low solubility of Zn due to the pH effect and also the phenomenon call ageing (Lock and Janssen, 2003). The trend of Zn solubility indicates slow dissolution of Zn minerals at the initial stage, and then the concentration dropped again due to the precipitation or ageing effect. Zinc concentrations were found to be low in all treatments using different rates of WTR (2.5, 5, 10, 20, and 40%) compared to the control (0% WTR). Addition of WTR did reduce the release of Zn from the sewage sludge. Therefore, WTR can be considered to be a potential soil amendment to fix Zn in contaminated soils.

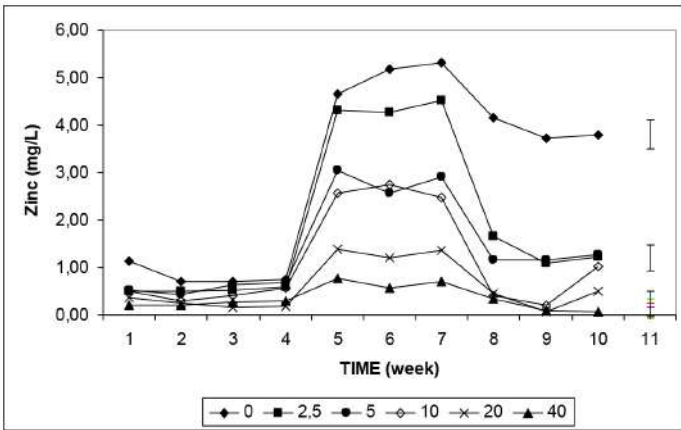
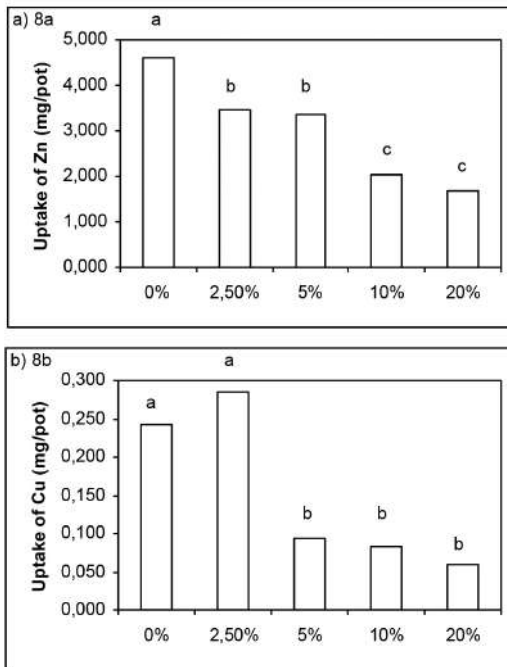


Figure 14 Soluble Zn at different rates of WTR treatments of contaminated soil.

Addition of WTR significantly reduced Zn uptake by corn plants compared to the control (Figure 15). This results show that the usage of WTR mixed with sewage sludge can significantly reduce the Zn uptake by corn. The major effect of high pH was to reduce the solubility of all micronutrients, especially Zn. Meanwhile, addition of more than 5% WTR, significantly reduced Cu uptake compared to the control. This results show that the usage of more than 5% WTR in sewage-sludge-amended soil can significantly reduce the Cu uptake by corn (Nur Hanani et al., 2008).



Similar letters above bars indicate that the values represented by the bars are not significantly different at the 1% level, according to the Duncan New Multiple Range Test (DMRT)

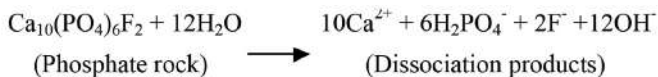
Figure 15 Uptake of trace metals, Zn and Cu (mg/pot) using soil amended with WTR

Phosphate materials

Phosphate compounds enhance the immobilization of metal(loid)s in soils through various processes including direct metal(loid) adsorption/substitution by P compounds, P anion-induced metal(loid) adsorption, and precipitation of metal(loid)s with solution P as metal(loid) phosphates. Depending on the source, soil application of P compounds can cause direct adsorption of metal(loid)s onto these compound through surface charge and enhanced anion-induced metal(loid) adsorption.

Phosphate rock (PR) belongs mainly to sedimentary, slightly to igneous and negligibly to metamorphic rocks. Eight percent of the world PR production is derived from the deposits of sedimentary marine origin, some 17% is derived from igneous rocks and their derivatives and the remainder comes from residual sedimentary deposits. About 90% of these deposits are used as raw materials for the manufacturing of phosphate fertilizer.

The dissolution of PR may be expressed by the equation;



Although the above reaction is for a fluorapatite PR, it applies to other members of the apatite minerals including reactive PR. As indicated in the above equation, the dissolution of PR results in the release of hydroxyl ions into the solution. Neutralization of the hydroxyl ions released by soil acidity enables the PR dissolution process to continue. Thus, an adequate supply of hydrogen ions is of primary importance for the continued dissolution of PR. Soil pH shows the magnitude of hydrogen ion supply, thus the use of PR depends on its reactivity and generally recommended for soils with a pH of 5.5 or less (Corley and Tinker, 2003).

Precipitation as metal(loid)-P has been proven as one of the main mechanism for the immobilization of metals such as Pb and Zn in soils. These fairly stable metal-P compounds have extremely low suitability over wide pH range, which make P application as attractive technology for managing metal(loid)-contaminated soils. Thus, application of apatite as amendment appears to be a promising soil additive for immobilizing metals in polluted soils (Soltan et al., 2012).

A glasshouse study has been conducted to assess the effectiveness of Pb immobilization due to chicken manure application using different sources of phosphate materials; bone meal (BM), Egyptian rock phosphate (ERP) and triple super phosphate (TSP) (Naim et al., 2017). From the soil fractionation study of a glasshouse experiment, the percentage of exchangeable fraction of Pb was reduced with application of P-amendments with the highest of 20.2% of reduction recorded for 2 t/ha application of TSP. This is followed by reduction in exchangeable fraction for others treatments: 2 t/ha of BM (4.1%), 4 t/ha of BM (5.1%), 1 t/ha of ERP (8.1%) and 2 t/ha of ERP at 17.6%. These treatments were recorded as being able to stabilize the Pb as indicated in the percentage reduction of phytoavailable pools into a more stable form of residual pool (Figure 17).

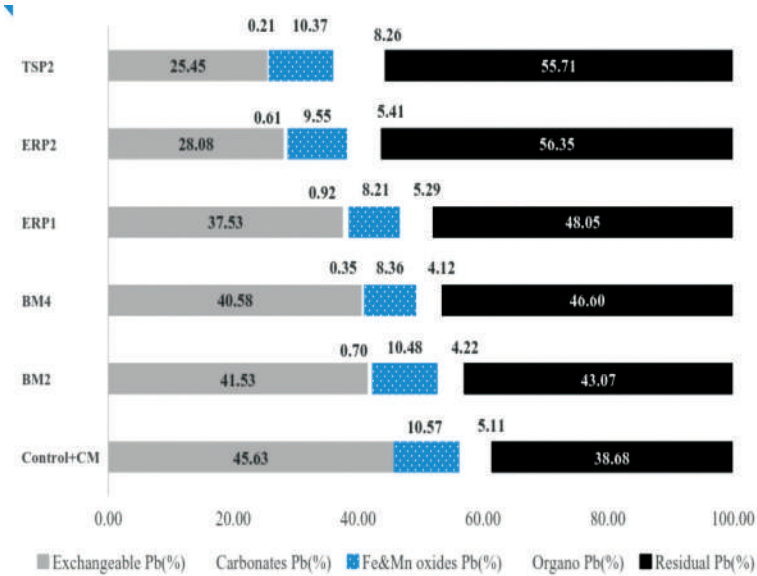
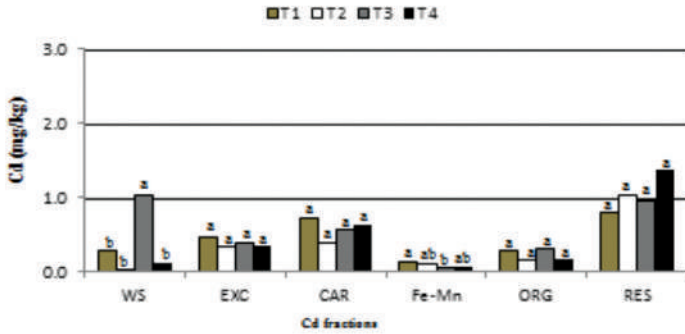


Figure 17 The lead content expressed as percentage in 5 fractions; 1) exchangeable, 2) carbonates, 3) Fe & Mn oxides, 4) organic matter and 5) residual with application of different P-amendments

Another glasshouse study was conducted to determine whether lime and palm oil mill effluent (POME) amendment at 4 different rates can help reduce Cd uptake by oil palm seedlings using Gafsa Phosphate Rock as a source of P fertilizer (Aini Azura et al., 2012).

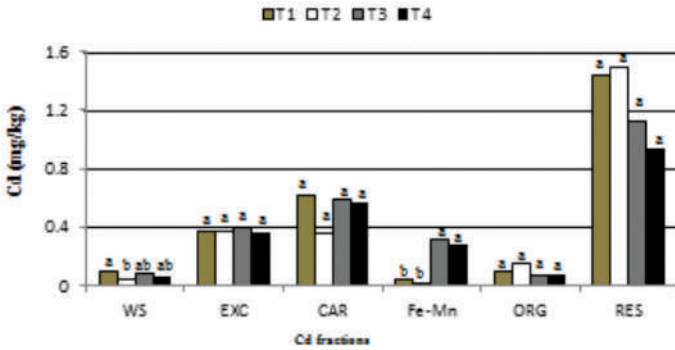
Segamat Series amended with POME showed significant differences ($p < 0.05$) of water soluble and Fe-Mn fractions between four rates of treatment (Figure 18). This treatment decreased the exchangeable whilst increasing the residual fraction, but because the amount was not substantial, the data were not statistically different. Meanwhile, cadmium in Segamat Series amended with lime was highest in the residual fraction followed by the exchangeable, carbonate, Fe-Mn, organic and lastly water soluble fractions (Figure 19).

Solid Waste Management



Letter with the same alphabet on the bars within the same soil fractions are not significantly different at $p > 0.05$. (Comparison made within rates of treatment)

Figure 18 Concentrations of Cd fractions in Segamat Series amended with four rates of POME



Letter with the same alphabet on the bars within the same soil fractions are not significantly different at $p > 0.05$. (Comparison made within rates of treatment)

Figure 19 Concentrations of Cd fractions in Segamat Series amended with four rates of lime

ISSUES PERTAINING TO IMMOBILIZATION TECHNIQUE

A major issue associated with immobilization technique is that although metal(loid)s become less available for plant uptake, their total concentrations in soils remain unchanged unless transported out of the soil system through leaching, colloid facilitated transport through surface runoffs, etc. The immobilized metal(loid)s may become plant available with time through natural weathering processes through the breakdown of high molecular weight metal(loid)s complexes or change in soil condition such as under waterlogged condition.

Most studies on immobilization of metal(loid)s are conducted on laboratory or glasshouse scale. More field studies are required to demonstrate the values of a range of immobilizing soil amendments to remediate contaminated soils. These field studies need to also examine the impact on the presence of co-contaminants, and also the long-term effectiveness of using soil amendments should be investigated.

CONCLUSION

There are potentials to use the industrial by-products as agricultural resources through land application. However, trace metal(loid)s in the by-products should be identified. Rates of application will be limited by trace metal(loid)s of concern and their presence in the environment should be monitored. There is also great potential of practising *in-situ* immobilization technique using soil amendments on agricultural soils that are not very high in metal(loid)s content. These amendments can reduce metal(loid)s uptake by the crops to below maximum permitted concentrations as gazetted in the

Malaysian Food Act (1983) and Food Regulations (1985) and thus the agricultural produce can be considered safe for human consumption.

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BIOGRAPHY

Che Fauziah binti Ishak was born on 7th of May 1959 in Bukit Kecil, Bukit Mertajam, Pulau Pinang. In 1977, she enrolled in University of Iowa, U.S.A. and graduated in 1981 with a Bachelor Degree in Chemistry. She continued her Masters of Science in Chemistry in 1981 at the same university. Upon returning to Malaysia, she started her career as a lecturer in 1985 at the Department of Soil Science (now known as the Department of Land Management), Faculty of Agriculture, Universiti Putra Malaysia. After serving for five years, she pursued her doctorate degree at the University of Georgia, Athens, Georgia, U.S.A. and obtained her PhD in Agronomy in 1993. She is currently serving as a Professor at the Department of Land Management, Faculty of Agriculture, Universiti Putra Malaysia and is an expert in the chemistry of heavy metals in the soil system.

To date, she has served for 32 years with UPM, fulfilling all the major responsibilities expected of her as a lecturer. She had earlier taught at the diploma and undergraduate levels, and later also including postgraduate courses. She has been actively involved in teaching the Basic Soil Science and Land Resources and Ecosystem for first degree courses, final year degree course in Environmental Soil Science, postgraduate courses in Soil and Plant Analysis and Land Contamination and Pollution. The experience gained through research as well as a deep interest in postgraduate work has led her to focus her effort and contribution to the smooth implementation of postgraduate research programmes and in the various administrative capacities (Head, Department of Land Management from 2007 -2010) and Deputy Dean (Academic, Student Affairs and Alumni) of Faculty of Agriculture from 2010 – 2016) she served at the university.

As a researcher, her subjects of interest are in contaminated soil with emphasis on heavy metals contamination of soils and assessment of land application of industrial by-products (mineral by-products and biosolids) on crop growth performance and soil fertility improvement, with special emphasis on heavy metals uptake by plants, which is becoming an important issue in food safety and is part of food security agenda. Her research also includes management of metal-contaminated soils through *in-situ* immobilization technique using industrial by-products such as red gypsum, coal fly ash, and water treatment residuals. To date she has had handled and completed six MOSTI funded research projects as the project leader and 15 research projects as a co-researcher, working on various aspects of soil chemistry and plant nutrition. She has also conducted a total of seven non-government funded project (contract research) such as with International Atomic Energy Agency (IAEA), Australian Centre of International Agricultural Research (ACIAR), Indah Water Konsortium (IWK), Huntsman Tioxide (M) Sdn. Bhd., CJ Bio Sdn. Bhd. and SKF Malaysia Sdn. Bhd. She hopes that the data generated during research can be utilized by policy and decision makers in the decision-making process and drawing up of policies pertaining to land application of these by-products.

She has been involved in soil, plant and fertilizer analyses activities ever since she joined UPM in 1985. She is currently the Coordinator of the Analytical Laboratory of the Department of Land Management, Faculty of Agriculture, UPM. She has been invited to give advice on the purchase of laboratory instruments and glasswares when RISDA's laboratory (ESPEK) was set up. She has also been invited to sit on the expert panel to evaluate fertilizers during RISDA's fertilizer tendering process. She has served as committee member of SIRIM Technical Committee on Fertilizers

and currently is a committee member of Malaysian Institute of Chemistry (MIC) Technical Committee on Fertilizers. Currently, she is the Chairman of SIRIM now known as MIC Technical Committee on Soil Quality. She has also been actively involved as a member of several professional societies such as Malaysian Society of Soil Science (MSSS) and is serving as Vice President from 2009 till at present, Chief Editor of Malaysian Journal of Soil Science (MJSS) from 2008 till at present, Management Committee of Agriculture Laboratory Association of Malaysia (AgLAM) from 2013 till at present.

She thrives for the best in whatever tasks she is entrusted with and excellence is her ultimate motive. She will continue to elevate herself towards greater achievements in the few years left as an academician in UPM.

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