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Heavy metals in soils and crops in southeast Asia.
1. Peninsular Malaysia

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Abstract

In a reconnaissance soil geochemical and plant survey undertaken to study the heavy metal uptake by major food crops in Malaysia, 241 soils were analysed for cation exchange capacity (CEC), organic carbon (C), pH, electrical conductivity (EC) and available phosphorus (P) using appropriate procedures. These soils were also analysed for arsenic (As), cadmium (Cd), cobalt (Co), chromium (Cr), copper (Cu), mercury (Hg), nickel (Ni), lead (Pb) and zinc (Zn) using *aqua regia* digestion, together with 180 plant samples using nitric acid digestion. Regression analysis between the edible plant part and *aqua regia* soluble soil As, Cd, Cr, Cu, Hg, Ni, Pb and Zn concentrations sampled throughout Peninsular Malaysia, indicated a positive relationship for Pb in all the plants sampled in the survey ($R^2 = 0.195$, $p < 0.001$), for Ni in corn ($R^2 = 0.649$, $p < 0.005$), for Cu in chilli ($R^2 = 0.344$, $p < 0.010$) and for Zn in chilli ($R^2 = 0.501$, $p < 0.001$). Principal component analysis of the soil data suggested that concentrations of Co, Ni, Pb and Zn were strongly correlated with concentrations of Al and Fe, which is suggestive of evidence of background variations due to changes in soil mineralogy. Thus the evidence for widespread contamination of soils by these elements through agricultural activities is not strong. Chromium was correlated with soil pH and EC, Na, S, and Ca while Hg was not correlated with any of these components, suggesting diffuse pollution by aerial deposition. However As, Cd, Cu were strongly associated with organic matter and available and *aqua regia* soluble soil P, which we attribute to inputs in agricultural fertilisers and soil organic amendments (e.g. manures, composts).

Introduction

The southeast Asian region is currently undergoing rapid development and extensive changes to the social and economic structure. Traditionally, agriculture has been the main base of the economies in this region but in recent years the industrial sector has achieved prominence. This has resulted in an increase in the size of urban populations and urban centres resulting in an increase in industrial and municipal waste. Many industrial plants in this region operate without any, or minimal, wastewater treatment and routinely discharge their waste into drains, which either contaminate rivers and streams or add to the contaminant load of biosolids (sewage sludge). Biosolids are increasingly being used as soil ameliorants and streams and rivers are the primary source of water for irrigation.

Contaminants can enter the food chain from industrial, urban and agricultural sources (Tiller *et al.* 1997). The contamination of agricultural soils and crops by heavy metals is causing concerns due to the potential effects on human health and the possible long-term sustainability of food production in contaminated areas. Residues in traded food commodities are also monitored by many countries and there are increasing incidences of trade in agricultural commodities being prohibited on the basis of contaminant concentrations. Where countries have conducted research to determine concentrations of contaminants in soil and food crops, regulations have been introduced to protect the soil resource from further contamination (McLaughlin *et al.* 1996). In Malaysia, there are currently no soil quality reference values for heavy metals (or toxic metals). Thus, there is a need to establish levels of heavy metals found in soils commonly used for agricultural production in Peninsular Malaysia (Khanif & Salmijah 1996).

In Malaysia, phosphatic fertilisers have been used for long periods in some regions. McLaughlin *et al.* (1996) have indicated that these fertilisers contain many impurities that can contaminate soils, and therefore it is important that the impact on soil and crop quality be assessed (Khanif & Salmijah 1996). Recent interest in the use of organic amendments as substitutes for the use of manufactured fertiliser has ignored the potential impacts from impurities in the new materials used.

Atmospheric pollution of soil is also increasing in Malaysia as urbanisation and industrialisation proceeds, yet few data are available to assess the impact and extent of the pollution.

The lack of any consistent investigation into contaminants in agricultural soils and crops in Malaysia make it difficult to identify potential problem areas. Thus there is a need to define the likely extent and severity of soil and crop pollution with inorganic contaminants.

This study was conducted with the aim of evaluating the normal ranges of heavy metals in agricultural soils of Peninsular Malaysia as well as the heavy metal levels in crops grown on these soils. These values will provide a baseline value for heavy metals in agricultural soils and therefore define first approximation reference values when evaluating soil contamination from different agricultural management practices such as the use of fertilisers, sewage sludge, industrial effluents, composts or farmyard manures.

Methodology

Soil and plant sampling and preparation

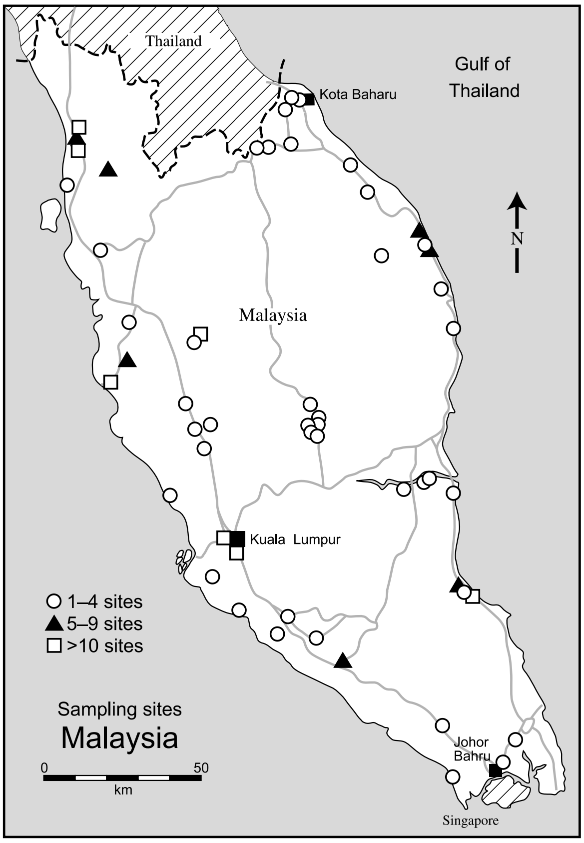

Fig. 1. Soil and plant sampling sites in Peninsular Malaysia.

A soil geochemical and concomitant plant survey to assess the extent of heavy metal pollution of soils and crops in Malaysia was based on 241 soil (0–15 cm) and 188 plant samples taken from agricultural production, forested and uncultivated regions (Figure 1). A reconnaissance survey approach using the main roads within a region (and not soil type) with sampling points approximately 25 km apart (Figure 1) was chosen as the basis of the investigation, since the level, source, and distribution of heavy metal pollution in Malaysia was unknown. Intensive agricultural areas were sampled more rigorously.

Soil samples from forested or uncultivated areas remote from industry were used as background values as these were assumed to be uncontaminated. Paired soil and edible plant parts were sampled in order to relate soil properties with the concentration of heavy metals in crops at each location. For each soil, five core samples were taken (50 mm diameter by 150 mm depth) with a stainless steel core sampler and combined into a composite sample for laboratory analyses. All samples were taken at least 75 m from any road to minimise contamination from automobile emissions and road dust. For horticultural and orchard samples, the soil cores were taken in between plants in the rows in which the plants were growing, over the length of a row. For forest and agricultural or paddy samples, the soil cores were taken around an approximately 20 m circle. The soils were air dried at ambient temperature and crushed to pass a 2 mm stainless steel sieve.

The edible portion of at least one plant was then sampled in close proximity to each soil core where available. Plants were only sampled if they were edible. Rice and agricultural products were taken from within the soil sampling area. Many soils were sampled without paired plants as either the plants had not reached maturity or a crop had not been planted but remnant plants indicated the previous year's crop. Plant samples were washed in reverse osmosis/deionised water, combined into a composite sample for laboratory analyses, oven dried at 70 °C and ground to pass a 1 mm stainless steel sieve.

The analytical values for cocoa, groundnut, mustard and rice are reported on a 'dry weight' basis since they are consumed in this form, while all other plants are reported on a 'fresh weight' basis.

Analyses

The soils were analysed for the following properties: pH, electrical conductivity (EC), cation exchange capacity (CEC), total organic carbon (%C), available P and *aqua regia* soluble Al, Fe, As, Cd, Co, Cr, Cu, Hg, Ni, Pb and Zn. Soil pH and EC ($\mathrm{dS\,m^{-1}}$) were determined on a 1:5 soil/water suspension. Cation exchange capacity was determined using the NH_4OAc at pH 7.0 leaching method of the Soil Survey Staff, Land Development Department (LDD) (1998). Available P was extracted from the soil by the Bray 2 method (0.03 N NH_4F and 0.1 N HCl, Bray & Kurtz 1945) using a 1:7 soil:solution ratio and determined using the ammonium molybdate/ascorbic acid method of Murphy and Riley (1962). Organic C was determined using a modified Walkley and Black procedure (Nelson & Sommers 1982). Soil Al, Fe and heavy metals were extracted in boiling *aqua regia* (Zarcinas *et al.* 1996) while plant heavy metals were solubilised by digestion with nitric acid (Zarcinas *et al.* 1987). Aluminium, Fe and the heavy metals, except Cd and Hg, in soils were determined by inductively coupled plasma spectrometry (ICPS) (SpectroFlame Modula, Spectro Analytical Instruments, Kleve, Germany). Cadmium in soils and plants, and Pb in plants were determined by electrothermal atomic absorption spectrometry (ETAAS), and Hg by cold vapour (CV) AAS (GBC Model 906, Melbourne, Australia).

The validity of the soil and plant digestion procedures and the ICPS operating parameters were established using internationally recognised National Institute of Standards and Technology (NIST) and National Research Council Canada Certified Reference Materials (CRC-CNRC) standard materials (Zarcinas *et al.* 1987, 1996). For analytical quality control, two in-house quality control samples (lettuce and spinach) and blanks were included in each plant analytical batch while two reference contaminated soils (State Chemical Laboratories, Victoria, Australia) and blanks were included in each soil analytical batch.

Statistical analyses

Statistical analysis and Principal Component Analysis (PCA), was performed using Genstat, Fifth edition, Release 4.2 (Lawes Agricultural Trust, Rothamsted, Harpenden, UK). Assessment of the normal distribution of the analytical data was based on the normal probability plot of residual log transformed data (derived from the regression analysis of log–log transformed data) versus sample percentile. Non-normal data were log-transformed to improve normal distribution and to reduce the influence of high analytical data. Correlation (r), analysis of variance (ANOVA) and regression analyses were performed on log-transformed soil analytical data.

PCA, based on the correlation matrix, was conducted for the soil chemical data set. The aim of using PCA was to ascertain any patterns in the soil samples in relation to these chemical characteristics, and hence infer possible relationships between these and other soil properties, fertiliser inputs or soil organic amendments.

Results and discussion

Data was evaluated by comparison of heavy metals with soil types (e.g. alluvial, sedimentary, sandy tailings from ex-tin mining areas or anthropogenic soils) and management practices. Management practices were assessed by comparing the concentration levels of cultivated soils against forested or uncultivated soils. In this case, the heavy metal concentrations of forested or uncultivated areas were considered as natural background values due to minimal anthropogenic influence.

For crop quality, the Maximum Permitted Concentration (MPC) as stated in the Malaysian Food Act (1983) and Food Regulations (1985) was used as a reference (Table 1). The ANZFA (1999) Australian Maximum Levels (MLs) and Generally Expected Levels

Table 1. Malaysian Food Act (1983) and Food Regulations (1985) (Fourteenth Schedule – Regulation 38) and Australian New Zealand Food Authority (1999, Proposal P157). Values in mg kg^{-1}. Malaysian data based on produce 'as consumed'. Australian data based on 'edible content of the food that is ordinarily consumed'. If dried, based on mass of food prior to drying.

Food	As		Cd		Cu[2]		Pb		Hg		Sb[2]		Sn[1]		Zn[2]	
	Mal	Aust	Mal	Aust	Mal	Aust	Mal	Aust	Mal	Aust	Mal	Aust	Mal	Aust	Mal	Aust
Vegetable product and fruit product other than vegetable juice and fruit juice	1	1	1	0.1	30		2	0.1	0.05		1		40		40	
Vegetable juice and fruit juice	0.1		1		10		0.5		0.05		0.15		40	250	5	
Tomato-pulp, paste and puree	2		1		100		2		0.05		1		40		40	
Tea, tea dust, tea extract and scented tea	1		1		150		2		0.05		1		40		40	
Coffee, chicory and related product	1		1		30		2		0.05		1		40		40	
Cocoa and cocoa product	1		1	0.5	70		2		0.05		1		40		40	

[1] All canned foods.

[2] Generally Expected Level (GEL).

(GELs) are also presented in Table 1. The GELs for metal contaminants in specific commodities are based on available data and reflect the generally expected levels in food commodities. As data for more commodities becomes available in the future, GELs will be established for these commodities. The Malaysian values were originally based on the values set by the New Zealand Food Authority and have not undergone any significant changes due to the lack of scientific data to justify any changes. The MPC was established on a food type 'as consumed' basis (personal communication, Ministry of Health of Malaysia).

Heavy metals, or trace metals, are a large group of trace elements, which are both industrially, and biologically important (Alloway 1995). At high concentrations heavy metals may become potentially toxic to humans, animals, plants or soil organisms. Heavy metals are found in all soils with the naturally occurring concentrations varying depending on the parent material from which the soil was formed. Heavy metals may be added to soils in agricultural fertilisers and pesticides, soil amendments (e.g. lime and gypsum), organic fertilisers (e.g. manures and composts), and in waste materials recycled to soil. In order to assess whether a soil has been contaminated by heavy metals, 'background' or benchmark values are needed against which a comparison can be made.

In order to establish soil 'background' heavy metal concentrations to assess the extent of urban soil contamination, Tiller (1992) recommended considering the range of values found in non-urban soils, preferably in regional agricultural, forest and virgin soils. However, Tiller (1992) also stated that due cognisance must be taken of metal impurities added with fertilisers and soil amendments, contaminants deposited from atmospheric dispersal, and differences due to metal variations in soil parent materials, which are reflected in the metal concentrations in the sampled soil. In Denmark, Scott-Fordsmand *et al.* (1996) used a '95% protection level' in order to establish a soil quality criterion to ensure protection of the terrestrial environment from the adverse effects of soil pollution. The Australian New Zealand Food Authority (ANZFA 1999) uses the 95th percentile as the most appropriate measure of high intake for commonly consumed food when using dietary models to establish health-based MPCs for metals. The criteria for 'good soil quality' described by Lamé and Leenaers (1998) is defined as the quotient of the 90th percentile of the background concentrations and the 'target value'. The target value allows for the simultaneous effect of a large number of heavy metals present in a contaminated soil containing 25% clay and 10% organic matter.

Table 2. Dutch target values and Australian ecological investigation levels (EIL) (mg kg^{-1}).

Metal	Dutch target value	Australian EIL
As	29	20
Cd	0.8	3
Cr (III)	100	400
Cu	36	100
Hg	0.3	1
Ni	35	60
Pb	85	600
Zn	140	200

The studies above established the percentile levels of heavy metals in order to define soil quality criteria for the protection of soil microorganisms and human health and the maintenance of the soil resource for plant production. In this study, the 95th percentile value of the randomly selected agricultural and background soils sampled for this study were used as the minimum concentration of a heavy metal for a soil to be considered contaminated. These 'investigation levels' provide a threshold value, which can trigger further evaluation of the possible contamination. Investigation levels do not indicate a potential hazard, but rather that further investigation is needed to determine if the contamination is related to anthropogenic activity and/or could develop into a risk to the environment or human health. For comparison, the 'target' values used in the Netherlands for soil protection (Lamé & Leenaers 1998) and the Australian Ecological Investigation Levels (EILs) (NEPM 1999) are presented in Table 2. The Dutch target values are based on natural (background) soil levels and on negligible risk concentrations. The Australian EILs for urban settings are based on considerations of phytotoxicity, ANZECC (1992) B levels, and soil survey data from urban residential properties in four Australian capital cities.

Metal distribution among soil orders

The mean, median, minimum, and maximum heavy metal and soil fertility concentrations of the Malaysian soils sampled with and without separation into soil Orders (Soil Survey Staff (LDD) 1998) are reported in Table 3. From this data set, the 95th percentile

Table 3. Trace element distributions in the Malaysian soils sampled in this survey and subdivided by soil Orders (Soil Survey Staff 1998).

Soil Order	EC (μS cm^{-1})	pH	%C	CEC (cmol kg^{-1})	mg kg^{-1}									
					Avail. P	As	Cd	Co	Cr	Cu	Hg	Ni	Pb	Zn
All Malaysian soils ($N = 241$)														
Mean	195	5.40	2.63	13.6	375	16.8	0.12	2.8	25.9	16.4	0.147	13.7	26.4	38.0
Median	111	5.07	1.72	9.5	156	8.5	0.09	1.5	23.1	10.6	0.100	8.6	21.9	32.7
Min	11	3.04	0.02	1.7	0.10	0.28	0.01	0.05	1.1	0.37	0.002	0.4	0.85	2.9
Max	1410	7.01	11.6	72.9	3590	280	2.02	38.7	72.7	114	0.860	73.5	90.0	137
Alfisols ($N = 4$)														
Mean	69	4.76	0.72	12.6	7.9	7.2	0.05	18.4	8.6	22.3	0.07	13.4	28.7	60.7
Min	40	4.39	0.46	6.1	0.41	1.7	0.03	0.56	0.50	18.0	0.06	1.4	3.8	14.7
Max	100	5.61	0.94	17.1	18.1	17.6	0.06	38.7	20.0	28.1	0.09	21.2	49.4	136
Anthropogenic soil (sand mine tailings) ($N = 12$)														
Mean	178	6.10	1.12	8.40	471	7.1	0.13	2.2	18.5	22.1	0.10	8.9	30.2	54.8
Min	58	5.11	0.70	1.68	163	1.2	0.03	0.49	8.9	8.5	0.04	4.8	5.7	20.9
Max	561	6.72	1.61	21.0	920	14.2	0.38	3.6	42.1	46.7	0.33	14.8	60.5	112
Entisols (flooded paddy) ($N = 7$)														
Mean	149	4.75	2.20	19.5	19.9	3.9	0.06	2.2	26.3	6.2	0.09	8.2	27.2	31.0
Min	42	4.41	1.67	2.89	12.4	3.1	0.03	1.1	15.2	3.7	0.03	6.0	22.0	20.5
Max	337	5.06	2.39	30.7	41.8	4.5	0.12	3.8	39.6	8.3	0.15	12.7	33.8	44.2
Entisols (other crops) ($N = 15$)														
Mean	242	4.97	1.77	8.4	125	14.6	0.07	0.91	16.1	5.8	0.07	20.5	15.5	24.8
Min	38	4.19	0.76	2.7	15.5	1.4	0.01	0.59	4.7	1.9	0.04	1.2	7.5	10.1
Max	789	5.82	3.21	13.6	370	61.5	0.20	1.6	30.7	15.8	0.17	37.4	21.9	93.3
Histosols (peat) ($N = 8$)														
Mean	300	4.73	9.3	n.d.	510	55.0	0.40	2.2	22.1	47.2	0.24	6.8	23.9	40.0
Min	90	3.04	4.3	n.d.	36.2	0.91	0.01	0.35	1.3	20.3	0.01	3.0	2.1	15.1
Max	1099	6.19	11.5	n.d.	1370	278	2.02	4.0	72.7	80.2	0.41	11.5	81.2	91.7
Inceptisols (Acid sulphate) ($N = 6$)														
Mean	326	4.97	1.88	19.7	29.8	16.4	0.06	1.9	26.5	10.1	0.12	8.1	29.0	27.6
Min	56	3.47	0.77	8.86	13.8	4.8	0.02	0.62	24.5	5.6	0.08	3.0	12.9	9.1
Max	510	6.37	3.03	27.6	97.7	39.3	0.14	2.8	30.4	12.7	0.21	11.2	36.3	39.1

Inceptisols (flooded paddy) ($N = 11$)														
Mean	78	4.80	2.30	24.9	29.8	10.9	0.09	2.2	27.7	10.9	0.06	13.2	28.7	25.6
Min	40	4.04	1.97	10.2	0.05	1.5	0.04	1.7	19.7	7.1	0.02	4.9	18.7	14.9
Max	108	5.09	3.45	64.5	84.9	36.6	0.18	4.0	35.4	20.1	0.09	73.5	50.8	41.3
Inceptisols (muck) ($N = 22$)														
Mean	215	5.41	4.20	14.3	519	12.4	0.17	1.9	22.8	20.8	0.20	8.7	28.4	33.1
Min	36	3.85	0.67	4.09	17.9	0.84	0.01	0.11	3.9	3.7	0.03	1.0	3.5	6.8
Max	1025	6.61	5.87	39.9	2940	49.9	0.40	5.3	44.9	38.8	0.86	16.9	56.8	57.5
Inceptisols (other) ($N = 8$)														
Mean	402	5.02	4.07	30.7	1340	34.5	0.17	2.5	31.7	37.6	0.10	12.6	26.3	58.1
Min	123	3.53	1.34	23.8	438	0.64	0.07	0.32	1.6	9.7	0.06	2.8	5.5	31.5
Max	1276	6.93	10.3	40.6	3590	61.5	0.25	3.7	48.7	114	0.20	19.3	41.4	103
Oxisols ($N = 41$)														
Mean	194	5.31	1.59	19.8	697	14.8	0.10	4.2	30.2	17.9	0.13	12.7	18.8	33.8
Min	11	3.43	0.42	2.19	4.1	2.2	0.01	0.31	3.7	1.3	0.01	0.4	2.3	9.6
Max	1375	6.85	4.95	50.3	2950	39.6	0.23	24.1	67.8	64.2	0.31	27.4	45.1	135
Background	*53*	*4.5*	n.d.	*93*	*2.2*	*13.1*	*0.01*	*0.44*	*46.1*	*8.9*	*0.14*	*7.0*	*38.9*	*24.3*
Spodosols ($N = 17$)														
Mean	162	5.18	1.57	6.28	246	1.7	0.06	0.21	4.9	8.0	0.06	2.2	6.4	11.6
Min	12	4.54	0.52	1.65	2.0	0.4	0.01	0.12	1.1	2.0	0.01	0.10	2.3	2.9
Max	1222	6.11	2.55	11.5	2950	4.7	0.15	0.34	13.0	16.5	0.17	6.0	15.3	21.2
Ultisols ($N = 58$)														
Mean	224	5.49	1.77	10.3	288	22.0	0.12	3.5	38.0	13.6	0.14	20.4	31.3	53.0
Min	29	4.04	0.22	3.8	0.05	0.28	0.02	0.04	0.10	0.37	0.10	0.91	0.85	2.9
Max	1409	7.01	4.48	72.9	1820	103	0.43	33.1	68.3	54.2	0.81	44.3	89.9	136
Background	*20*	*4.57*	*0.02*	*3.8*	*16.3*	*2.4*	*0.02*	*10.7*	*2.8*	*4.7*	*0.01*	*52.0*	*88.6*	*33.3*
Assoc. of Ultisols and Oxisols ($N = 27$)														
Mean	86	5.10	1.28	10.3	208	6.7	0.08	1.1	17.8	6.9	0.14	19.7	36.8	31.1
Min	20	4.08	1.03	2.91	8.6	1.8	0.01	0.39	2.8	2.3	0.06	3.9	6.9	11.8
Max	389	6.38	1.76	35.4	1590	15.6	0.38	2.3	52.9	19.1	0.30	64.9	85.8	73.6
Background	*20*	*5.07*	*0.59*	*2.9*	*5.2*	*83.1*	*0.02*	*0.31*	*2.8*	*14.4*	*0.03*	*1.9*	*10.1*	*9.9*
Ultisols (Acid sulphate) ($N = 3$)														
Mean	144	4.74	n.d.	n.d.	18.2	8.9	0.02	1.5	19.3	3.7	0.15	5.5	20.0	18.5
Min	36	4.60	n.d.	n.d.	17.3	4.0	0.02	1.1	12.9	2.8	0.10	5.1	9.8	12.9
Max	348	4.95	n.d.	n.d.	19.2	17.3	0.03	2.2	25.9	5.1	0.20	5.8	28.9	27.0

n.d. – not determined.

Table 4. The 95% 'Investigation Levels' determined for 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

investigation levels, for the heavy metals determined in this study, were established (Table 4).

Background and spodosol soil samples all had significantly lower heavy metal concentrations than soils sampled from the other agricultural areas (ANOVA, F pr. < 0.001). This indicates heavy metal contamination to the majority of agricultural soils in Peninsular Malaysia due to anthropogenic activity (possibly added in fertilisers, wastes, pesticides, effluents or atmospheric sources) that may develop into a risk to the environment or human health. The spodosols are possibly low in heavy metals due to their seasonal flooded environments resulting in metal depletion either by drainage or leaching.

When considered by soil Order, the soils most contaminated with heavy metals were the Inceptisols and Ultisols (ANOVA, F pr. < 0.001). These represent 40% of the soils sampled reflecting their dominance as agricultural soils, and therefore probable extensive fertiliser history with chemical fertilisers and/or organic amendments. The metal content of the Histosols may also reflect the metal content in the original organic materials.

When considered by sub-groups, the sandy textured Spodosols, the acidic (sulphatic) and reducing (sulphidic – flooded) Inceptisols and Ultisols, and the flooded paddy Entisols all had lower concentrations of heavy metal (ANOVA, F pr. < 0.001). This may be a reflection of low fertiliser and organic amendment input into these marginal agricultural soils or leaching of some of the contaminant metals from the root zone.

The Histosols and Ultisols, predominantly used for high value vegetable and fruit production, had the highest concentration of Cd (ANOVA, F pr. < 0.001). These soils also had the highest soil P concentrations (ANOVA, F pr. < 0.001). The high Cd concentration in the Histosols may also be an indication of elevated Cd in the natural organic material.

Heavy metals in soils and crops

The great majority of cultivated soils of Peninsular Malaysia are heavily fertilised with phosphatic fertilisers as evidenced by the difference in available P concentrations between agricultural and non-agricultural soils (Table 3). The addition of phosphatic fertilisers to the agricultural soils has resulted in a concomitant increase in As, Cu, Cd, and Zn (Table 5). Regression analysis resulted in highly significant log *aqua regia* soluble soil As (Figure 2, $R^2 = 0.721$, $p < 0.001$), Cd (Figure 3, $R^2 = 0.801$, $p < 0.001$), Cu (data not shown, $R^2 = 0.363$, $p < 0.001$), and Zn (Figure 4, $R^2 = 0.537$, $p < 0.001$) versus log aqua *regia* soluble P relationships.

The non-transformed linear *aqua regia* soluble soil As and Cd versus *aqua regia* soluble P concentration regression confirmed the low background soil As and Cd concentrations and the addition of As and Cd as a contaminant of phosphatic fertilisers. The non-transformed curvilinear *aqua regia* soluble soil Cu and Zn versus *aqua regia* soluble P concentration regression is indicative of the variably high background Cu and Zn concentrations (soil P values below 1000 mg kg^{-1}) and the addition of Cu (and some Zn) as contaminants of phosphatic fertilisers (figures not shown).

Correlation analysis between the log of the *aqua regia* soluble heavy metal concentrations for 241 soils sampled (Table 6) showed a positive correlation with log of the *aqua regia* soluble P, Al and Fe, available P, and log % soil C. These correlations imply the addition of heavy metals with phosphatic fertilisers and organic soil amendments, and an association with indigenous clay minerals in the soil.

The PCA of the soil samples from Malaysia (Figure 5) show that the first two principal components of 25 assessed account for 34.7% of the overall variability in the data (PC1 – 19.0% and PC2 – 15.7%). *Aqua regia* soluble soil Co, Ni, Pb, and Zn are highly correlated with soil Al and Fe. Therefore, either soil contamination has not occurred due to agricultural activities and therefore these elements are associated with indigenous clay minerals in the soil, or that contamination has been associated with soils types that are higher in Al and Fe. This latter hypothesis is probable, as soils high in Al and Fe have high fertiliser P

Table 5. Trace element distribution (mg kg^{-1} except plant Hg µg kg^{-1}) in soils and corresponding crops of Peninsular Malaysia.

Plant	As		Cd		Cr		Cu		Hg		Ni		Pb		Zn	
	Soil	Plant	Soil	Plant	Soil	Plant	Soil	Plant	Soil	Plant	Soil	Plant	Soil	Plant	Soil	Plant
Background ($N = 5$)																
Mean	21		0.016		21		8		0.063		13		32		17	
Min	1		0.014		1		4		0.006		1		2		3	
Max	83		0.02		46		14		0.136		52		89		33	
Angled loofa ($N = 6$)																
Mean	5	0.033	0.1	0.006	16	0.14	11	0.35	0.079	0.2	7	0.07	14	0.14	29	1.4
Min	2	0.033	0.044	0.002	4	<0.01	4	0.19	0.014	0.075	2	0.03	6	0.028	17	1
Max	9	0.058	0.19	0.017	39	0.02	20	0.55	0.176	0.27	17	0.15	31	0.28	45	2
Brinjal ($N = 9$)																
Mean	39	0.089	0.09	0.021	30	0.025	22	0.82	0.218	0.3	11	0.13	27	0.007	42	1.7
Min	4	0.07	0.01	0.013	4	0.021	2	0.2	0.173	0.05	1	<0.03	8	0.0008	10	1.3
Max	200	0.168	0.19	0.039	64	0.029	67	1.5	0.26	0.1	22	0.19	45	0.019	92	2.1
Cabbage ($N = 8$)																
Mean	22	0.079	0.19	0.013	31	0.06	28	3.6	0.15	0.5	25	0.12	47	0.021	60	3
Min	4	0.06	0.01	0.002	5	<0.01	2	<0.01	0.02	0.05	3	<0.03	11	0.0005	10	0.5
Max	39	0.17	0.35	0.046	64	0.227	49	20.3	0.33	1.9	45	0.26	90	0.065	136	6.1
Chilli ($N = 25$)																
Mean	13	0.157	0.09	0.011	19	0.036	15	1.78	0.13	0.74	12	0.24	24	0.11	37	3.2
Min	1	0.14	0.01	0.001	2	<0.01	2	0.61	0.02	0.05	1	0.11	3	0.0005	7	2.2
Max	64	0.36	0.38	0.033	65	0.093	64	14.2	0.41	5.4	65	0.42	82	0.15	112	5.1
Cocoa ($N = 5$)																
Mean	30	1.21	0.11	0.666	35	0.49	11	14.1	0.16	15	13	4.5	26	0.29	39	52
Min	5	<1	0.02	0.204	21	0.02	5	4.9	0.05	2.9	6	2.5	18	0.10	10	40
Max	71	2.69	0.17	1.68	52	1.41	17	26.1	0.25	39	30	8.8	38	1.2	93	77
Corn ($N = 10$)																
Mean	26	0.042	0.12	0.001	44	0.024	25	0.18	0.16	0.16	20	0.06	31	0.0021	38	1.5
Min	3	0.04	0.08	0.001	21	<0.01	7	<0.01	0.05	0.05	9	<0.03	15	0.0005	16	0.6
Max	61	0.08	0.16	0.002	65	0.178	41	0.25	0.31	0.39	28	0.11	49	0.010	63	2.2
Cucumber ($N = 13$)																
Mean	25	0.059	0.06	0.006	15	0.018	18	0.34	0.12	0.25	7	0.1	18	0.014	41	2.4
Min	1	0.04	0.02	0.002	2	<0.01	4	0.32	0.03	0.22	2	0.07	4	0.012	6	2
Max	280	0.117	0.15	0.012	48	0.021	58	0.38	0.41	0.3	12	0.13	49	0.019	113	3.1

Table 5. (*continued*)

Plant	As		Cd		Cr		Cu		Hg		Ni		Pb		Zn	
	Soil	Plant	Soil	Plant	Soil	Plant	Soil	Plant	Soil	Plant	Soil	Plant	Soil	Plant	Soil	Plant
Groundnut ($N = 4$)																
Mean	17	<1	0.08	0.178	45	0.256	10	9.6	0.14	1.8	8	2.6	16	0.11	24	40
Min	16	<1	0.03	0.06	36	0.142	4	5.6	0.12	0.5	5	1.1	10	0.032	15	33
Max	18	<1	0.19	0.281	50	0.45	26	11.5	0.2	5.4	14	6.5	33	0.32	37	45
Lady's finger ($N = 9$)																
Mean	28	0.12	0.19	0.026	33	0.039	27	0.79	0.18	0.34	17	0.18	31	0.0061	33	5.6
Min	9	0.09	0.03	0.001	10	<0.01	5	0.17	0.05	0.05	4	<0.03	14	0.0005	13	3.6
Max	87	0.22	0.29	0.066	68	0.147	44	1.35	0.31	0.96	28	0.25	75	0.030	47	8.4
Long bean ($N = 19$)																
Mean	14	0.089	0.13	0.002	20	0.024	18	0.96	0.13	0.41	11	0.25	27	0.0052	37	4.7
Min	1	0.08	0.04	0.001	2	<0.01	3	0.47	0.01	0.05	2	0.12	4	0.0005	7	3.4
Max	103	0.173	0.4	0.005	66	0.045	54	1.74	0.29	0.1	38	0.52	58	0.022	70	6.5
Mustard ($N = 10$)																
Mean	16	<1	0.09	0.25	26	1.4	15	6.1	0.11	17.5	15	1.7	21	0.31	30	73
Min	1	<1	0.02	0.06	4	0.37	3	3.7	0.01	7.4	2	0.04	5	0.062	10	34
Max	48	<1	0.19	0.7	67	6	35	8.9	0.27	55.1	46	3.1	69	0.72	52	121
Oil palm ($N = 6$)																
Mean	12	<0.54	0.11	0.006	22	1.34	4	4.1	0.14	6.1	9	1.3	15	0.12	17	2.8
Min	1	<0.54	0.02	0.002	4	0.82	1	1.8	0.08	0.5	1	1	2	0.0005	10	1.2
Max	30	<0.54	0.38	0.012	54	2.3	6	6	0.23	31.8	36	1.7	33	0.75	40	7.4
Rice ($N = 16$)																
Mean	8	1.27	0.07	0.011	27	0.37	9	1.9	0.07	2.8	12	1.1	28	0.24	28	42
Min	1	<1	0.03	0.004	15	0.036	4	0.51	0.02	0.05	5	0.6	19	0.10	15	16
Max	37	2.59	0.18	0.04	40	2.7	20	3.1	0.16	6	74	3.5	51	0.70	44	265
Spinach ($N = 8$)																
Mean	31	0.081	0.28	0.023	33.5	0.17	22.3	0.59	0.089	0.001	40.3	0.17	65.1	0.034	108	9.6
Min	5.5	0.07	0.055	0.009	12.6	0.017	18.6	0.30	0.010	0.0005	8.5	0.04	10.3	0.001	25.8	5.0
Max	48	0.14	0.36	0.099	68	0.48	68	0.86	0.169	0.0016	84	0.32	100	0.11	120	28.9
Water convolvulus ($N = 6$)																
Mean	49	0.28	0.12	0.026	32	0.13	20	0.8	0.15	1.5	10	0.18	22	0.034	35	3.9
Min	1	<0.09	0.01	0.007	5	0.03	3	0.17	0.03	0.6	3	<0.03	9	0.0090	7	2.8
Max	202	0.9	0.23	0.055	73	0.31	37	1.6	0.27	2.3	24	0.28	49	0.10	59	7.7

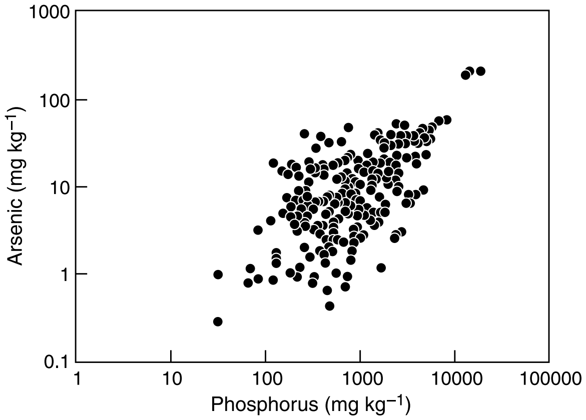

Fig. 2. Regression (log As = log (0.5646 × P) + log(−0.7143) of log *aqua regia* soluble arsenic versus log *aqua regia* soluble phosphorus for the Malaysian soils sampled in this survey ($R^2 = 0.721$, $p < 0.001$).

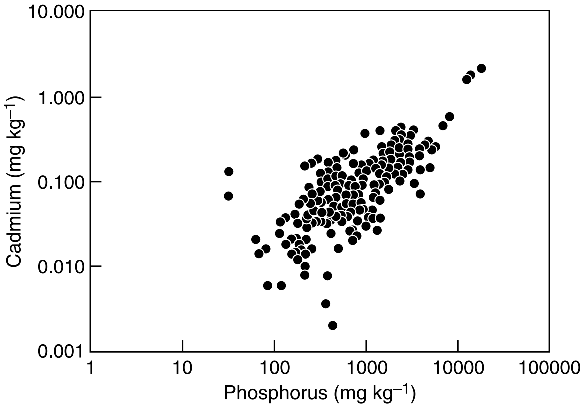

Fig. 3. Regression (log Cd = log (0.6603 × P) + log (−2.9998) of log *aqua regia* soluble cadmium versus log *aqua regia* soluble phosphorus for the Malaysian soils sampled in this survey ($R^2 = 0.801$, $p < 0.001$).

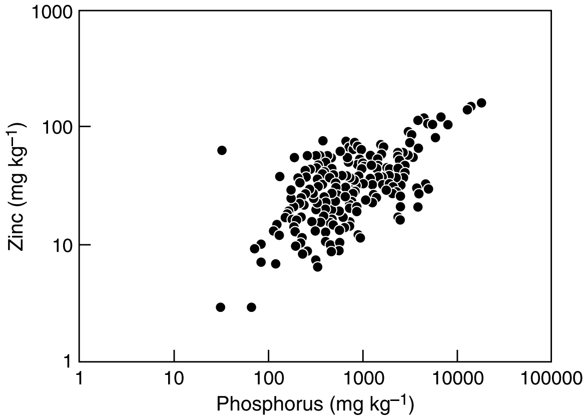

Fig. 4. Regression (log Zn = log (0.3892 × P) + log (0.3538) of log *aqua regia* soluble zinc versus log *aqua regia* soluble phosphorus for the Malaysian soils sampled in this survey ($R^2 = 0.537$, $p < 0.001$).

requirements, so that histories of P fertiliser addition, with associated impurities, are likely to be greater on these soils. However, *aqua regia* soluble and available P concentrations were not correlated with Al and Fe, lending weight to the argument that fertiliser addition to these soils was not generally high, and the association of Co, Ni, Pb, and Zn with Al and Fe concentrations was related to variations in background mineralogy.

Aqua regia soluble soil As, Cd, and Cu are highly correlated with soil organic C and P, and were enriched in many agricultural soils suggesting sources related to agricultural inputs.

Aqua regia soluble soil Cr is highly correlated with soil pH, Ca, Na, S and EC suggesting the association with soil salinity and acid sulphate soil components, while *aqua regia* soluble soil Hg was not correlated with any soil matrix or agricultural input component, suggesting diffuse pollution by aerial deposition.

Arsenic

The concentration of As in plant edible portions is below the Malaysian MPC for all crops except cocoa, groundnut, mustard and rice (Table 5). Water convolvulus (morning glory) and oil palm seed pulp had the highest As concentrations of the fresh weight plants, while there were no significant differences for the dry weight plants (ANOVA, F pr. < 0.001). This was indicative of the low bioavailability of As in the soil resulting in a low transfer coefficient of As to the plant tissue (Smith *et al.* 1998). The brinjal (aubergine), lady's fingers, groundnut and water convolvulus soils contained the highest concentrations of As (ANOVA, F pr. < 0.001) (Table 6). The reason for the high concentration of soil As is not known. The high concentration of As in water convolvulus would indicate this plant to be an accumulator of As. Using the Dutch target value for soil protection and the Australian EIL (Table 2) the majority of the agricultural soils (Table 5) are contaminated with As. These high As levels may be due to arsenopyrite which is known to occur in many regions of southeast Asia, especially in tin mining regions.

Cadmium

Cadmium, usually added to the soil as an impurity of phosphatic fertiliser, is the heavy metal of most concern, as its transfer from soil to the edible portions of agricultural food crops is significantly greater than for other contaminant elements (McLaughlin *et al.* 1996). Although only cocoa had maximum

Table 6. Correlation (r) matrix of the log of soil fertility parameters and the log of aqua *regia* soluble metals in the Malaysian soils sampled in this survey, $N = 241$.

	log Avail P	log %C	log Al	log As	log Cd	log Co	log Cr	log Cu	log Fe	log Hg	log Ni	log P	log Pb	log Zn
log Avail P	1													
log %C	*0.173*	1												
log Al	−0.101	*−0.140*	1											
log As	*0.124*	*0.216*	*0.497*	1										
log Cd	*0.429*	*0.414*	*0.310*	0.408	1									
log Co	0.112	−0.080	*0.481*	0.361	0.322	1								
log Cr	−0.050	0.089	*0.706*	0.637	0.361	0.392	1							
log Cu	*0.272*	*0.492*	*0.185*	0.357	0.576	0.435	0.299	1						
log Fe	*−0.219*	*−0.177*	0.680	*0.559*	*0.157*	*0.573*	*0.619*	0.104	1					
log Hg	*−0.162*	*0.446*	*0.405*	0.352	0.219	0.010	0.388	0.228	0.254	1				
log Ni	−0.021	*−0.140*	*0.699*	0.472	0.357	0.584	0.681	0.349	*0.563*	0.177	1			
log P	0.721	*0.294*	*0.228*	*0.465*	*0.661*	*0.409*	*0.267*	*0.603*	*0.145*	0.088	*0.356*	1		
log Pb	0.008	−0.076	*0.688*	0.403	0.278	0.671	0.484	0.310	*0.495*	0.124	0.714	*0.318*	1	
log Zn	*0.286*	0.096	*0.469*	0.384	0.551	0.672	0.373	0.541	*0.492*	0.156	0.561	*0.578*	0.597	1

Significant at the 0.05 probability level when $r \geq 0.124$.
Significant at the 0.01 probability level when $r \geq 0.162$.
Significant at the 0.001 probability level when $r \geq 0.206$.
Italics indicates significant correlations between environmentally important parameters.

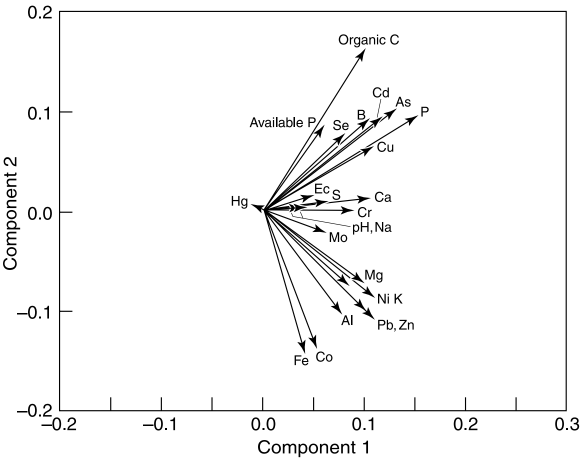

Fig. 5. PCA of the heavy metal and fertility parameters for the Malaysian soils sampled in this survey.

concentrations that exceeded the Malaysian MPC of 1 mg kg^{-1} (Table 5) and had the highest concentration of Cd in the plants sampled (ANOVA, F pr. < 0.001), groundnut and mustard seeds (*Brassica* sp.) had Cd values that exceed the Australian MLs (Table 1). The majority of the agricultural soils (Table 5) were below the Dutch target value for soil protection and the Australian EIL (Table 2).

Cadmium may also be detrimental to the soil microbial population. Chaudri *et al.* (1992) demonstrated that a long time of exposure was required (18 months) in order to observe a total elimination of *Rhizobium* in soil as a result of Cd contamination at a rate of 6 mg kg^{-1} in a single dose. Smaller decreases in *Rhizobium* numbers were shown to occur at lower Cd concentrations (2 mg kg^{-1}). Although there were no surveyed soils at this level of Cd, the cumulative addition of Cd via phosphatic fertilisers and especially industrial wastes, may have a detrimental effect on the soil microorganisms at significantly lower levels of Cd than those reported. The effect of heavy metals on soil microorganisms was not assessed in this study.

Chromium

Oil palm seed pulp had the highest Cr concentrations of the fresh weight plants while mustard seeds had the highest Cr concentrations for the dry weight plants (ANOVA, F pr. < 0.001) (Table 5). Chromium (as CrIII) is an essential nutrient in animal based agricultural systems while CrVI has been shown to be carcinogenic. There are no international guideline values for Cr in foods and all the agricultural soils (Table 5) were below the Dutch target value for soil protection and the Australian EIL (Table 2).

Copper

Cabbage and oil palm seed pulp had the highest Cu concentrations of the fresh weight plants (ANOVA, F pr. < 0.05) while cocoa had the highest Cu concentrations for the dry weight plants (ANOVA, F pr. < 0.001)

(Table 5). No crops exceeded the Malaysian MPC for Cu (Table 1). The highest cabbage and chilli Cu concentrations were likely due to the use of copper-based fungicides, which is usually resident on the surface of plant tissues, but can be washed off resulting in high soil Cu levels (Table 5). Many of the agricultural soils (Table 5) are above the Dutch target value for soil protection while only one soil growing pineapple (data not presented) exceeded the Australian EIL (Table 2). Copper is strongly attached to organic material and may have been added as a contaminant with organic soil amendments.

There is also now a considerable body of evidence documenting a decrease in the soil microbial biomass as a result of the long-term exposure to heavy metal contamination (e.g. Cu) from past applications of sewage sludge (McGrath 1994), Cu and Zn from animal manures (Christie & Beattie 1989), and past applications of Cu-containing fungicides (Zelles *et al.* 1994). These decreases in microbial biomass can occur at relatively low metal loadings (Dahlin *et al.* 1997). The moderate levels of Cu found during this survey and the continuing addition of Cu via Cu-based fungicides and industrial wastes may have a detrimental effect on the soil microorganisms at significantly lower levels of Cu than those reported in the literature cited above.

Mercury

Spinach and water convolvulus had the highest Hg concentrations of the fresh weight plants (ANOVA, F pr. < 0.05) while cocoa and mustard seed had the highest Hg concentrations for the dry weight plants (ANOVA, F pr. < 0.006) (Table 5).

Maximum concentrations of Hg in mustard seed exceeded the Malaysian MPC (Table 1) while cocoa and oil palm seed pulp were 75% of the MPC. Many of the agricultural soils (Table 5) were above the Dutch target value for soil protection but below the Australian EIL (Table 2).

Nickel

Oil palm seed pulp had the highest Ni concentrations of the fresh weight plants (ANOVA, F pr. < 0.001) while cocoa and groundnut had the highest Ni concentrations for the dry weight plants (ANOVA, F pr. < 0.001) (Table 5).

Only the soils on which chilli and rice were grown did the Ni values exceed the background values (Table 5). This is a reflection of the naturally occurring variable background Ni levels, with many tropical soils high in Ni. There are no international guideline values for Ni in foods. Four of the agricultural soils (Table 5) were above the Dutch target value for soil protection but only one soil growing rice was above the Australian EIL (Table 2).

Lead

Oil palm seed pulp had the highest Pb concentrations of the fresh weight plants (ANOVA, F pr. < 0.001) while there was no statistical difference in Pb concentrations between the dry weight plants (ANOVA) (Table 5).

It is unlikely that the high soil Pb concentrations determined during this survey are due principally to Pb emissions from vehicles using leaded petrol as the soils were sampled at least 75 m from any road, but the reason for the high concentration of soil Pb is not known. Lead levels of crops were generally low. No crop exceeded the Malaysian MPC (Table 1). Two of the agricultural soils (Table 5) exceeded the Dutch target value while no soil exceeded the Australian EIL (Table 2).

Zinc

Spinach had the highest Zn concentrations of the fresh weight plants (ANOVA, F pr. < 0.01) while there was no statistical difference in Zn concentrations between the dry weight plants (ANOVA) (Table 5).

Maximum concentrations of Zn in cocoa, groundnut, mustard, and rice exceeded the Malaysian MPC (Table 1). No agricultural soil (Table 5) exceeded the Dutch target value or the Australian EIL (Table 2). The high Zn values for the soils growing cabbage and cucumber were probably due to the use of zinc-based fungicides, which is usually resident on the surface of plant tissues, but can be washed off resulting in high soil Zn levels. However, these soil values were not translated into elevated plant concentrations.

Many researchers, particularly McGrath (1994), have shown that plant tissue nitrogen (N) levels and the percentage of soil N derived from fixation in clover, due to the symbiotic N_2-fixation by *Rhizobium* strains, were considerably reduced when the clover was grown on a soil contaminated with Zn, which was close to the prevailing European Union guideline for soil protection ($300\,\mathrm{mg\,kg^{-1}}$). It was shown that the clover rhizobia were far more sensitive to the toxic effects of heavy metals than were their host plants and this toxicity to free-living rhizobia in the soil resulted in their gradual extinction.

Relationships between heavy metals in plants and soils

Soil and climatic factors, and agronomic management govern the mobility and bioavailability of a metal in the soil. Uptake by plants and translocation of heavy metals to the edible portion is governed by the plant genotype. The distribution of heavy metals within the soil matrix is also governed by the type of clay minerals and organic matter present (Blaser & Zimmermann 1993).

Generally, *aqua regia* soluble heavy metal concentrations in soil are considered poor predictors of heavy metal bioavailability. Simple regression analysis between the edible plant parts As, Cd, Cr, Cu, Hg, Ni, Pb and Zn concentrations and *aqua regia* soluble soil concentrations indicated a significant positive relationship for Pb in all plants sampled in the survey ($R^2 = 0.195$, $p < 0.001$), for Ni in corn ($R^2 = 0.649$, $p < 0.005$), for Cu in chilli ($R^2 = 0.344$, $p < 0.010$), and for Zn in chilli ($R^2 = 0.501$, $p < 0.001$) (data not shown). Only the Ni regression had leveraging data. Exclusion of the leveraging data from the regression analysis resulted in a non-significant relationship. The significant relationship between edible plant part and *aqua regia* soluble soil Pb is a surprising result due the low mobility of contaminant Pb in soil and low bioavailability to plants (McLaughlin *et al.* 1996). However, these data indicate that as soil Pb pollution increases, the concentrations of Pb in the edible parts of plants (as assessed in this survey) may increase. The good relationships between soil and chilli Cu and Zn concentrations suggest chilli (*Capsicum annum*) may potentially be used as an indicator of soil pollution for these elements.

Conclusions

We suggest that the 95th percentile heavy metal concentrations can be used as 'Investigation Levels' for Peninsular Malaysia. The concentration ranges (minimum up to 95th percentile) for all Peninsular Malaysian topsoils sampled were As (0.28–56.7), Cd (0.01–0.32), Co (0.05–6.7), Cr (1.1–60.9), Cu (0.37–47.3), Hg (0.002–0.362), Ni (0.4–41.3), Pb (0.85–65.8) and Zn (2.9–92.0) mg kg^{-1}. Soils with element concentrations above these investigation levels are likely to have been contaminated due to the addition of metals in fertilisers, wastes, pesticides, effluents or atmospheric sources. These values should be used to trigger further investigation of the reasons/sources for the contamination of the soil and an assessment made of the potential risks to humans or the environment if the contamination continues.

Cobalt, Ni, Pb and Zn were associated with Al and Fe suggesting these elements were intrinsically associated with the soil matrix. Arsenic, Cd, and Cu were associated with *aqua regia* soluble and available P, and organic matter suggesting these metals are associated with agricultural inputs, for example, composts and chemical fertilisers. Chromium was correlated soil pH and EC, Ca, Na and S 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.

The natural levels of As, Cr and Ni in many southeast Asian soils are significantly higher than European soils and therefore exceed soil contamination guideline values in many European countries. This highlights the dilemma in extrapolating data across regions and emphasises the need to establish metal contamination guideline values for individual regions.

Cocoa, groundnut, mustard and rice had elevated concentrations of all the metals assessed when compared to the other plants. One sample of spinach contained the highest Zn while one sample of cabbage contained the highest Cu level. This may be due to the widespread use of Cu and Zn pesticides on these crops.

As heavy metals in soils persist indefinitely, the concentration to which heavy metals are allowed to accumulate in soils should be set conservatively. Based on the toxic effect of Zn on *Rhizobium*, the United Kingdom Government revised the recommendations for metal accumulation in soils receiving biosolids from 300 to 200 mg Zn kg^{-1} (MAFF/DoE 1993). As the minimum loadings of heavy metals to soil at which negative effects are observed become smaller (Dahlin *et al.* 1997), decisions on 'acceptable' risk of damage to soil microorganisms and microbial processes will need to be revised.

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