ASSESSMENT OF HEAVY METAL CONCENTRATIONS IN DIFFERENT SOIL TYPES AND VEGETABLES GROWN IN MUAR, JOHOR

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By

NOORHAFIZAH BINTI RAHIM

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

April 2006

DEDICATION

I would like to dedicate this thesis especially to:

My beloved parent,

Rahim bin Abdullah

and

Siti Hawa binti Mohd Yatim

Sisters,

Norazlina
Azean Musfirah

& lastly my Husband

Nurul Hasyrin bin Halim

Who always supported and encourage me to do the best.

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Master of Agricultural Science

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April 2006

Chairman: **Associate Professor Siti Zauyah Bte Darus, PhD**

Faculty: **Agriculture**

Large areas of land in Muar district, Johor, are especially used for vegetable cultivation. Four major cultivation areas were selected for this study namely, Solok, Pagoh, Sawah Ring and Sengkang which had different types of soils. In these areas, 53 sampling points with a total of 159 soil samples comprising of 66 samples of organic clay and muck (OCM), 42 samples of Holyrood series, 27 samples of Durian series and 24 samples of Jerangau series were collected from the vegetable cultivation areas. The soils were sampled at 0-20 cm, 20-40 cm and 40-60 cm depths. From each of the sampling areas, one sample of vegetable was collected thus, making a total of 53 vegetable samples. Forty-three samples of background topsoils (0-20 cm) from each location that were considered as unaffected or minimally affected by man were sampled.

The soil samples were analyzed for pH, organic carbon (OC), cation exchange capacity (CEC), texture and mineralogy. The aqua-regia method was used to determine the total heavy metal concentrations and three extractants i.e. ethylenediaminetetraacetic acid (EDTA), diethylenetriaminepentaacetic acid (DTPA) and 0.1 M hydrochloric acid (HCl) were used to extract the available heavy metal concentrations. The chemical partitioning of heavy metals in cultivated mineral and background topsoils were determined using Tessier's sequential extraction. Dry ashing method was used to determine the contents of heavy metals in vegetable samples. Flame Atomic Absorption Spectrophotometry (AAS) was used to analyze all heavy metals (Cd, Cr, Cu, Ni, Pb and Zn) that were extracted from soil and vegetable samples.

Statistically, the pH for all of the soil series showed no significant differences at $p \leq 0.05$ with depths. Organic clay and muck and Durian series had a significantly higher OC contents in topsoils than subsoils. There were no significant differences for OC contents in Holyrood and Jerangau series with depths. The differences of CEC between soil depths were not statistically significant ($p \leq 0.05$) for Holyrood, Durian and Jerangau series but were statistically significant for OCM, between 0-20 cm and 40-60 cm depths.

The total heavy metal concentrations in cultivated topsoils showed a significant increase as compared to the background topsoils. The ranges of total Cd, Cr, Cu, Ni, Pb and Zn concentrations in the cultivated soils were 0.06-3.72 mg kg^{-1}, 0.14-77.8 mg kg^{-1}, 0.74-128.0 mg kg^{-1}, 0.2-258.0 mg kg^{-1}, 0.02-39.6 mg kg^{-1} and Zn 6.2-

208.2 mg kg^{-1}, respectively. Generally, total concentrations of Cd, Cr, Cu and Zn were significantly higher in the topsoils of OCM and Holyrood series than Durian and Jerangau series. Most of total heavy metal concentrations in OCM and Holyrood series showed significant differences with depths. Chromium, Pb and Zn in Durian series showed significant differences with depth whereas in Jerangau series, only Cr and Pb showed significant differences with depth.

In the cultivated mineral topsoils, Cd, Cu, Ni, Pb and Zn were dominated in the residual fraction and were least present in Fe-Mn oxide for Cd, Cr and Cu whereas Ni, Pb and Zn were least present in the organic fraction. The percentages of Ni, Pb and Zn in the residual fraction of cultivated mineral topsoils were much higher than the background mineral topsoils.

The total Zn had a good correlation with pH (r = 0.74, n = 22), total Pb and Cr with CEC (r = 0.95**, n = 8; r = 0.51*, n = 22). No correlations were found between total heavy metal concentrations in soil and heavy metal uptake by vegetables. Cadmium and Pb in vegetables showed positive correlation with CEC (r = 0.77**, n = 14; r = 0.54**, n = 22) whereas Pb and Zn in vegetables showed positive correlation with OC (r = 0.63*, n =14; r = 0.60*, n = 14). Zinc in vegetables also showed positive correlation with pH (r = 0.73*, n = 9). Available Cu in soil extracted by DTPA was positively correlated with Cu (r = 0.44*, n = 22) in vegetable and available Cd extracted by 0.1 M HCl with Cd (r = 0.84*, n = 8) in vegetables.**

Most of the heavy metals in the cultivated and background topsoils were below the 95th percentile and Dutch standard, except for Cd. Among the soil types, topsoils of OCM and Holyrood series clearly showed that Cd had exceeded both limits whereas Zn was only above 95th percentile value. The heavy metal contents in the leafy vegetables were significantly higher than fruit vegetables, except for Cr on fresh weight basis. The heavy metal contents in vegetables were still below the maximum permitted concentration (MPC) that was allowed by Malaysian Food Act (1983) and Food Regulation (1985).

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

PENILAIAN KEPEKATAN LOGAM BERAT DALAM PELBAGAI JENIS TANAH DAN SAYURAN YANG DITANAM DI MUAR, JOHOR

Oleh

NOORHAFIZAH BINTI RAHIM

April 2006

Pengerusi: **Profesor Madya Siti Zauyah Bte Darus, PhD**

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Sebahagian besar tanah di daerah Muar, Johor digunakan bagi penanaman sayur. Empat kawasan penanaman sayur utama telah dipilih untuk kajian ini iaitu Solok, Pagoh, Sawah Ring dan Sengkang yang mempunyai jenis tanah yang berbeza. Daripada kawasan tersebut, sebanyak 53 titik persampelan dengan jumlah 159 sampel tanah yang terdiri daripada 66 sampel tanah lumpur dan lempung organik (LLO), 42 sampel siri Holyrood, 27 sampel siri Durian dan 24 sampel siri Jerangau telah diambil daripada kawasan tanaman sayur. Tanah-tanah tersebut disampel pada kedalaman 0-20 sm, 20-40 sm dan 40-60 sm. Daripada setiap kawasan

persampelan tanah, satu sampel sayur diambil menjadikan 53 sampel sayur kesemuanya. Empat puluh tiga sampel tanah lapisan atas latarbelakang dari setiap lokasi yang dianggap tidak terganggu dan kurang dipengaruhi oleh aktiviti manusia disampel pada kedalaman 0-20 sm.

Sample-sampel tanah dikaji untuk menentukan pH, karbon organik (KO), keupayaan pertukaran kation (KPK), tekstur dan mineralogi. Kaedah aqua-regia digunakan untuk menentukan jumlah kepekatan logam berat dan tiga kaedah pengekstrakkan iaitu asid atelindiamintetrasetik (EDTA), asid dietalinatriaminpentasetik (DTPA) and 0.1 M asid hidroklorik (HCl) digunakan untuk mengekstrak kepekatan logam berat tersedia. Pembahagian kimia logam berat dalam tanah lapisan atas mineral tanaman sayur dan tanah lapisan atas latarbelakang ditentukan melalui kaedah pengekstrakkan pemeringkatan *Tessier*. Kaedah pengabuan kering digunakan untuk menentukan kandungan logam berat dalam sampel sayur. Kesemua logam berat (Cd, Cr, Cu, Ni, Pb dan Zn) yang diekstrak daripada sampel tanah dan sayur dianalisa oleh Spektrofotometer Serapan Atom Nyalaan.

Kandungan karbon organik bagi tanah LLO dan siri Durian menunjukkan perbezaan yang sangat bererti pada lapisan tanah atas berbanding tanah lapisan bawah. Tiada perbezaan bererti bagi kandungan KO dalam siri Holyrood dan Jerangau dengan kedalaman tanah. Perbezaan KPK antara kedalaman tanah tidak bererti ($p \leq 0.05$) bagi siri Holyrood, Durian dan Jerangau tetapi terdapat

perbezaan bererti pada nilai KPK bagi lumpur and lempung organik antara kedalaman 0-20 sm dan 40-60 sm.

Jumlah kepekatan logam berat bagi tanah lapisan atas tanaman sayur adalah sangat bererti berbanding lapisan tanah atas latarbelakang. Julat jumlah kepekatan bagi Cd, Cr, Cu, Ni, Pb dan Zn dalam tanah tanaman sayur masing-masing adalah 0.06-3.72 mg kg^{-1}, 0.14-77.8 mg kg^{-1}, 0.74-128.0 mg kg^{-1}, 0.2-258.0 mg kg^{-1}, 0.02-139.6 mg kg^{-1} dan 6.2-208.2 mg kg^{-1}. Secara keseluruhan, kepekatan Cd, Cr, Cu dan Zn adalah sangat bererti dalam lapisan atas tanah LLO dan siri Holyrood berbanding siri Durian dan Jerangau. Kebanyakan logam berat yang terdapat dalam tanah LLO dan siri Holyrood menunjukkan perbezaan bererti dengan kedalaman tanah. Kromium, Pb dan Zn dalam siri Durian menunjukkan perbezaan bererti dengan kedalaman tanah manakala hanya Cr dan Pb dalam siri Jerangau menunjukkan perbezaan bererti dengan kedalaman.

Dalam tanah lapisan atas mineral, Cd, Cu, Ni, Pb dan Zn didominasi oleh bentuk sisa baki dan rendah dalam oksida Fe-Mn bagi Cd, Cr dan Cu manakala Ni, Pb dan Zn rendah dalam bentuk organik. Peratusan Ni, Pb and Zn dalam sisa baki tanah lapisan atas mineral adalah lebih tinggi berbanding tanah lapisan atas latarbelakang.

Jumlah Zn mempunyai hubungan yang baik dengan pH ($r = 0.74^{**}$, $n = 22$), jumlah Pb dan Cr dengan KPK ($r = 0.95^{**}$, $n = 8$; $r = 0.51^{*}$, $n = 22$). Didapati tiada korelasi didapati antara jumlah kepekatan logam berat dalam tanah dan logam

berat yang diambil oleh sayur. Kadmium dan Pb dalam sayur menunjukkan korelasi positif dengan KPK (r = 0.77, n = 14; r = 0.54**, n = 22) manakala Pb dan Zn dalam sayur menunjukkan korelasi positif dengan KO (r = 0.63*, n =14; r = 0.60*, n = 14). Zink dalam sayur juga menunjukkan korelasi positif dengan pH (r = 0.73*, n = 9). Kuprum tersedia dalam tanah yang diekstrak oleh DTPA mempunyai korelasi positif dengan Cu (r = 0.44*, n = 22) dalam sayur dan Kd tersedia yang diekstrak oleh 0.1 M HCl dengan Cd (r = 0.84*, n = 8) dalam sayur.**

Kebanyakan logam berat dalam tanah lapisan atas tanaman sayur dan tanah lapisan atas latarbelakang adalah di bawah 95th persentil and piawaan Dutch, kecuali Cd. Antara lapisan atas siri-siri tanah, LLO dan siri Holyrood menunjukkan bahawa nilai min Cd adalah melebihi kedua-dua piawaan manakala hanya Zn melebihi nilai 95th persentil. Kandungan logam berat dalam sayur daun adalah sangat bererti berbanding dalam sayur buah, kecuali Cr dalam berat basah. Kandungan logam berat dalam sayur masih di bawah nilai kepekatan penyerapan maksimum yang dibenarkan oleh Akta Makanan Malaysia (1983) dan Peraturan Makanan (1985).

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I certify that an Examination Committee has met on 24 April 2006 to conduct the final examination of Noorhafizah binti Rahim on her Master of Agricultural Science thesis entitled "Assessment of Heavy Metal Concentrations in Different Soil Types and Vegetables Grown in Muar, Johor" in accordance with Universiti Pertanian Malaysia (Higher Degree) Act 1980 and Universiti Pertanian Malaysia (Higher Degree) Regulations 1981. The Committee recommends that the candidate be awarded the relevant degree. Members of the Examination Committee are as follows:

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DECLARATION

I hereby declare that the thesis is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that is has not been previously or concurrently submitted for any other degree at UPM or other institutions.

NOORHAFIZAH BINTI RAHIM

Date:

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LIST OF ABBREVIATIONS

AAS	**Atomic Absorption Spectrophotometry**
CEC	**Cation exchange capacity**
DMRT	**Duncan's multiple range test**
DTPA	**Diethylenediaminetetracetic acid**
EDTA	**Ethylenediaminetetra acetic acid**
GIS	**Global Information System**
GPS	**Global Positioning System**
HCl	**Hydrochloric Acid**
MPC	**Maximum Permitted Concentration**
NH_4OAc	**Ammonium acetate**
OC	**Organic carbon**
SAS®	**Statistical Analysis System**