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Metal Contamination of Vegetables Grown on Soils Irrigated with Untreated Municipal Effluent

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Abstract Metals in soils and vegetables irrigated with untreated municipal/industrial effluent, from four cities of Pakistan (Gujranwala, Sialkot, Hyderabad and Mirpurkhas) were assessed. The cadmium, copper, lead and chromium concentrations in the municipal/industrial effluent from all sites were above the recommended permissible limits. Similarly, cadmium, lead and nickel concentrations in almost all the soil samples were above the recommended permissible limits with chromium higher than the recommended permissible limits in 62% soils and copper higher in 26%. Cadmium and chromium concentrations were above the recommended permissible limits in all the examined vegetables and lead was exceeded in 90% of vegetables.

Keywords Metals · Municipal/industrial effluent · Soils · Vegetables

Growing of vegetables in peri-urban areas is a common practice in many Asian and African countries. Such crops are invariably irrigated with untreated municipal/industrial wastewater (Gupta et al. 2008) because of its availability, low cost, high concentration of organic components and some nutrient value. However, its excessive and long-term application to arable land can adversely affect soil health and groundwater quality since it contains considerable amount of metals such as cadmium (Cd), chromium (Cr), lead (Pb), nickel (Ni), copper (Cu) (Mapanda et al. 2005).

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Most of the developed countries have permissible limits either for maximum metal load in soils or the amount of sewage sludge/wastewater and trace metals concentration in the sewage sludge/wastewater being applied to soils (Keller et al. 2002). However, in developing countries including Pakistan, applications of sewages sludges are being applied to agricultural land without bothering about their quality and quantity. This results in elevated concentration of metals and other pollutants in surface horizons (Ghafoor et al. 1995; Hussain et al. 2006). Majority of soils, developed from alluvial deposits in South Asia, are low in organic matter and cation exchange capacity and hence are low in metal retention. When the capacity of the soil to retain metals is overloaded/diminished due to repeated use of wastewater, soils release metals into soil solution which can either be leached to shallow groundwater or taken up by plants. Frequently the quantities of metals accumulated by plants are large enough to cause clinical problems both to animals and human beings if they are consumed. For example accumulation of excessive metals in the human body can cause carcinogenic, cardiovascular and gastrointestinal diseases.

In this study, Cd, Cr, Pb, Ni and Cu concentration in municipal/industrial effluent, used for irrigation, pre-urban soils and vegetables, irrigated with untreated municipal/industrial effluent, were assessed. The levels of contamination were evaluated with respect to international food standard guidelines.

Materials and Methods

Municipal/industrial effluent used for irrigation without any pre-treatment, soil (under wastewater irrigation) and vegetables were collected from peri-urban area of

Gujranwala (32°09′ N and 74°11′ E), Sialkot (32°29′ N and 74°32′ E), Hyderabad (25°22′ N and 68°21′ E) and Mirpurkhas (25°31′ N and 69°01′ E), Pakistan to measure Cd, Cr, Pb, Ni and Cu concentrations.

A total of 37 municipal/industrial effluent samples were collected from municipal disposal stations in 250 mL polypropylene sampling bottle and treated with distilled nitric acid. The effluent samples were filtered through 0.45 μm cellulose nitrate filter. A total of 56 soil and 156 vegetable samples were collected by selecting a transect of 5 × 5 m at random at each site. From each transect, a composite soil sample was taken from 4 thoroughly mixed subsamples taken at random locations within the transect area. The soil samples were air-dried, ground and passed through 2 mm sieve. A portion of ≈100 g was drawn from the 2 mm fraction and reground to obtain <200 μm fraction. The randomly sampled edible portions of vegetable (e.g., leafy, root, fruit) from each transect were brought to the laboratory and were pass through a three step washing sequence (Reuter et al. 1986). The washed samples were cut into small pieces; air and oven (at 70°C) dried and ground.

Soil samples were analyzed for different physical and chemical parameters following the standards procedures described by Sparks et al. (1996) and Dane and Topp (2002). The filtrates of digested samples of soils and vegetables and sludge/wastewater samples were analyzed for metals using a graphite furnace atomic absorption spectrometer (Perkin Elmer AAnalyst 800). Detection limits for Cd, Cr, Pb, Ni and Cu are 0.8, 3.0, 15.0, 6.0 and 1.5 μg L^{-1}, respectively.

Results and Discussion

Soil organic matter concentrations at Gujranwala (0.83–2.03%), Sialkot (0.52–1.69%), Hyderabad (1.24–2.0%) and Mirpurkhas (0.83–1.92%) were adequate in surface soils but less in subsurface soils. The soils were alkaline in reaction (pH—7.2 to 8.6) and non-saline (EC—0.11 to 1.77 dS m^{-1}). The soils of Gujranwala and Sialkot were less calcareous ($CaCO_3$—0.5–15.2%) than the soils of Hyderabad and Mirpurkhas ($CaCO_3$—5.3 to 24.5%). The cation exchange capacity (CEC) varied between 7.9 and 15.1 meq/100 g in surface soils and 8.2 and 15.8 meq/100 g in subsurface soils.

Metal analyses of untreated municipal effluents used for irrigation in Gujranwala, Sialkot, Hyderabad and Mirpurkhas peri-urban areas are summarized in Table 1. The range of Cd concentration (μg L^{-1}) was 11–23, 6–17, 1–60 and 6–14 in effluent collected from Gujranwala, Sialkot, Hyderabad and Mirpurkhas, respectively. In comparison with the FAO/WHO (2001) standard guideline for wastewater irrigation, more than two-thirds of samples were above the permissible limit (10 μg Cd L^{-1}). Copper concentrations in almost all the effluent samples were also above the permissible limit of 17 μg Cu L^{-1}. The highest Cu concentrations (2 μg to 1,133 μg Cu L^{-1}) were observed in effluents collected from Gujranwala. Similarly, Pb and Cr concentration in all the effluent samples exceeded the recommended levels of 65 μg Pb L^{-1} and 550 μg Cr L^{-1} except at Hyderabad where Cr concentration in all samples was within the safe limit. Surprisingly, Ni contents in the all samples collected from the four cities were within safe limits. The high concentrations of metals in the effluents collected from all the cities is most probably due to the indiscriminate disposal of untreated municipal and cartage industrial wastewater to agricultural lands, canals, rivers and drains (Hussain et al. 2006). These high metal concentrations confirmed the earlier reported composition by Hussain et al. (2006). Similar compositions of sewage effluent are common in developing countries like India (Gupta et al. 2008) and Nigeria (Oguzie and Okhagbuzo 2010).

The concentration of Cd (mg kg^{-1} of dry soil) in soils of the study areas ranged from 1.6 to 9.5 in surface soil and 1.2–8.9 in sub-surface soil (Table 2). The maximum Cd concentrations were observed at Mirpurkhas (5.2 in subsurface and 9.5 mg kg^{-1} surface soil) and all of the collected soil samples exceeded the permissible limit

Table 1 Metals concentrations of municipal/industrial effluent used for irrigation (μg L^{-1}) in the study areas

Element	Site							
	Gujranwala		Sialkot		Hyderabad		Mirpurkhas	
	Range	Means	Range	Means	Range	Means	Range	Means
Cd	11–23	17 (100[a])	6–17	11.4 (72)	1–60	19 (63)	6–14	10.7 (67)
Cu	2–1,133	131 (93)	6–676	75.8 (87)	19–54	39 (100)	22–50	41 (100)
Pb	21–588	221 (79)	12–419	135.5 (62)	113–454	327 (100)	120–440	247 (100)
Cr	613–7,255	1,176 (87)	15–19,870	2,021 (78)	177–510	297 (0)	1,185–1,329	1,258 (100)
Ni	10–3,438	404 (7)	9.8–2,178	392 (7)	1–90	33 (0)	5–30	17 (0)

[a] Percentage of samples above FAO/WHO (2001) recommended permissible limits

Table 2 Total metal concentrations (mg kg^{-1}) of soils irrigated with municipal/industrial effluent

Elements	Sites							
	Gujranwala		Sialkot		Hyderabad		Mirpurkhas	
	Depths (cm)							
	0–15 (34[a])	15–30 (34)	0–15 (22)	15–30 (22)	0–15 (12)	15–30 (12)	0–15 (8)	15–30 (8)
Cd								
Range	1.6–8.8	1.2–8.9	2.9–6.9	1.4–6.9	2.2–5.7	2.0–5.0	6.7–9.5	5.2–8.4
Means	5.9 ± 1.8	5.1 ± 2.1	4.5 ± 1.2	3.8 ± 1.6	3.9 ± 1.2	3.3	8.3 ± 1.2	7.2 ± 1.5
Above RPL* (%)	93	79	92	60	78	56	100	100
Cu								
Range	38.5–379.3	31.5–174.6	13.5–106.6	12.9–72.9	24.0–62.6	13.5–41.0	19.4–38.5	13.4–31.5
Means	169.5 ± 80.3	105.1 ± 38.4	53.6 ± 32.5	35.7 ± 17.2	37.7 ± 13.7	26.6	26.64 ± 7.4	20.3 ± 6.9
Above RPL (%)	89.7	51.7	4.0	0	0	0	0	0
Pb								
Range	78.5–519.3	37.6–332.1	66.2–283.4	42.75–279.0	36.4–131.6	25.2–71.0	125.8–226.4	121.7–219.7
Means	163.6 ± 89.0	102.5 ± 75.7	134.7 ± 69.9	109.7 ± 82.0	72.3 ± 30.0	47.9 ± 5.0	182.6 ± 42.3	155.3 ± 37.6
Above RPL (%)	79.3	34.5	44	40	11.1	0	100	100
Cr								
Range	80.6–304.8	45.06–203.4	62.77–323.3	48.75–137.1	33.2–106.1	20.6–86.9	122.9–151.7	104.0–143.3
Means	159.4 ± 67.8	102.9 ± 57.7	107.5 ± 48.9	80.5 ± 25.8	49.9 ± 25.1	35.3 ± 6.0	134.6 ± 11.3	122.3 ± 19.3
Above RPL (%)	89.7	34	44	20	11	0	100	100
Ni								
Range	42.5–233.2	15.48–132.0	36.9–146.5	26.2–158.6	51.7–65.3	38.9–54.1	80.8–112.9	63.2–101.0
Means	104.7 ± 46.0	71.2 ± 41.5	81.7 ± 33.7	74.7 ± 40.1	55.1 ± 4.3	47.4 ± 3.0	99.9 ± 13.4	86.6 ± 15.3
Above RPL (%)	89.7	58.6	92	72	100	44.4	100	100

* Recommended permissible limits

[a] Number of samples

(3 mg kg^{-1}). Khan et al. (1994) also reported elevated total Cd concentrations in calcareous soils irrigated with municipal effluent. Higher Cd concentrations in calcareous soils exceeding the threshold values have also been reported in other developing countries like India (Gupta et al. 2008; Sharma et al. 2007) and Zimbabwe (Mapanda et al. 2005).

The total Cu concentrations in all soils of the study areas except Gujranwala were within the permissible limits of 100 mg kg^{-1} despite the municipal effluent used for irrigation having a Cu concentration greater than the permissible limits. In Gujranwala, Cu concentrations ranged from 38.5 to 379.3 mg kg^{-1} in surface soil and 31.5–174.6 mg kg^{-1} in subsurface soil. Although the Cu concentrations of Sialkot, Hyderabad and Mirpurkhas soils were within the permissible limits, these were several fold higher than those reported by Gupta et al. (2008) and Oguzie and Okhagbuzo (2010).

The highest mean Pb concentrations (182.6 ± 42.3 mg kg^{-1}) were observed in soils of Mirpurkhas, ranging between 122 and 226 mg kg^{-1} and all samples were above the permissible limits (100 mg kg^{-1}), while 79% of soils at Gujranwala, 44% at Sialkot and 11% at Hyderabad were above the permissible limit. The results from this study are on the higher side of the range reported by Gupta et al. (2008) and Sharma et al. (2007) for calcareous soils. An almost similar trend was observed for total soil Cr concentrations.

The total soil Ni concentrations were higher than the permissible limit (50 mg kg^{-1}) in almost all soils from the four sites. The total mean Ni concentration in surface soils was in the order Gujranwala < Mirpurkhas < Sialkot < Hyderabad. The difference in mean Ni concentration between surface and sub-soil was smaller than reported by Kachenko and Singh (2006). However, Ni concentrations observed in this study were several fold higher than those reported by Sharma et al. (2007) for the urban fringe area of Varanasi, India.

There were large variations in the concentrations of metals in soil among sites. This variation may be correlated with soil properties such as pH, conductivity, clay content, organic matter content, and calcareousness as well as additions of metal through untreated municipal/industrial effluent. Specific adsorption of metals onto organic matter, clay minerals, humic acid and fulvic acid are other possible

Table 3 Metal concentrations (mg kg^{-1}) in vegetables grown in soils irrigated with municipal/industrial effluent

Site/vegetable	Cadmium		Copper		Lead		Chromium		Nickel	
	Range	Means	Range	Means	Range	Means	Range	Means	Range	Means
Gujranwala										
Bitter gourd (n* = 8)	0.1–1.1 (5[a])	0.6 ± 0.4	16–60	21 ± 17	8–27 (8)	16 ± 5	1–27 (3)	8.1 ± 11	3.2–20.6	12.1 ± 6.0
Brinjal (n = 5)	0.4–2.3 (5)	1.4 ± 0.9	8–88	17 ± 9	2–23 (5)	14 ± 12	0.1–8 (1)	2.2 ± 3.3	0.6–16.1	8.4 ± 7.3
Cauliflower (n = 8)	0.1–0.5 (6)	0.4 ± 0.2	4–50	18 ± 17	0.3–27 (6)	8 ± 12	7–13 (8)	11 ± 2	5.1–18.9	11.1 ± 5.6
Chillies (n = 12)	0.1–3.3 (11)	0.9 ± 0.8	6–30	13 ± 8	0.1–35 (9)	23 ± 12	0.1–9.2 (5)	2.8 ± 2.3	4.1–18.8	12.6 ± 5.3
Coriander (n = 9)	0.4–2.0 (9)	1.2 ± 0.5	11–129 (2)	55 ± 53	23–58 (9)	37 ± 16	9.7–28 (8)	19.6 ± 7.4	4.3–20.4	8.7 ± 5.4
Gourd (n = 8)	0.03–1.0 (7)	0.6 ± 0.3	6–32	15 ± 9	1.2–48 (8)	13 ± 15	0.1–15.4 (4)	5.2 ± 5.6	5.7–18.8	10.8 ± 9.8
Okra (n = 9)	0.1–1.1 (8)	0.7 ± 0.4	5–21	11 ± 6	3.3–30 (9)	13 ± 9	0.9–18 (4)	5 ± 5.8	6.5–19.8	10.4 ± 4.1
Lettuce (n = 6)	0.5–1.2 (6)	0.9 ± 0.4	16–49	28 ± 17	12–37 (6)	23 ± 11	17–22 (6)	20.4 ± 2.4	9.6–12.4	10.9 ± 1.3
Spinach (n = 13)	0.9–4.3 (13)	2.8 ± 1.2	10–40	24 ± 9	15–90 (9)	38 ± 10	5.0–22.1 (4)	14.6 ± 6.5	8.4–21.9	12.9 ± 4.3
Sponge gourd (n = 10)	0.05–1.0 (8)	0.6 ± 0.2	6–25	18 ± 6	0.2–36.4 (9)	14 ± 11	0.1–10.7 (4)	2.4 ± 3.2	7.0–17.2	11.8 ± 3.2
Sialkot										
Bitter gourd (n = 4)	0.5–1.6 (4)	0.9 ± 0.6	4.3–8.6	6.3 ± 1.8	2.7–28.8 (4)	16.0 ± 11.5	0.1–14.2 (1)	4.4 ± 6.6	5.0–21.5	14.0 ± 7.6
Brinjal (n = 4)	0.4–0.7 (4)	0.5 ± 0.1	9.1–10.9	10.2 ± 0.9	0.2–26.8 (4)	17.6 ± 11.9	1.0–11.3 (1)	3.8 ± 5	3.9–25.1	11.8 ± 9.2
Chillies (n = 6)	0.7–1.5 (6)	1.2 ± 0.3	2.8–18.5	10.2 ± 5.0	2.8–26.3 (6)	15.6 ± 9.8	0.6–18.7 (2)	5.6 ± 7.1	3.6–26.8	11.1 ± 8.4
Coriander (n = 4)	1.2–2.9 (3)	2.0 ± 0.8	7.5–11.9	9.7 ± 2.2	12.9–26.3 (3)	20.8 ± 7.1	2.1–5.9 (3)	3.9 ± 1.9	5.6–8.3	7.1 ± 1.4
Cucumber (n = 7)	0.6–2.4 (7)	1.0 ± 0.7	2.7–18.3	7.8 ± 6	0.5–30.4 (7)	13.4 ± 13.1	1.7–20.9 (4)	8.9 ± 8.4	4.4–20.3	13.2 ± 6.7
Gourd (n = 7)	0.3–1.1 (7)	0.6 ± 0.3	3.3–11.0	7.8 ± 2.8	0.7–29.9 (7)	19.0 ± 10.0	0.8–17.6 (3)	5.3 ± 6.9	2.8–28.6	10.5 ± 9.6
Okra (n = 10)	0.6–1.9 (10)	1.1 ± 0.5	3.5–11.8	7.5 ± 3.3	0.1–22.6 (8)	9.8 ± 8.9	0.9–20.1 (7)	7.8 ± 7.3	3.1–37.4	16.7 ± 11.2
Turnip (n = 3)	0.5–0.6 (3)	0.5 ± 0.1	3.5–6.6	4.7 ± 1.6	3.9–6.5 (3)	5.2 ± 1.3	5.5–6.7 (3)	6.1 ± 0.6	8.9–10.0	9.4 ± 0.6
Hyderabad										
Bottle gourd (n = 4)	0.7–5.6 (3)	2.4 ± 1.6	7.3–18.0	12.7 ± 5.4	13.2–42.5 (3)	25.4 ± 13.3	0.2–1.8 (2)	1.0 ± 0.6	2.8–19.4	9.3 ± 6.2
Cauliflower (n = 3)	0.1–8.4 (2)	3.0 ± 1.7	7.5–18.3	13.8 ± 5.7	7.5–18.3 (3)	13.8 ± 5.7	3.0–11.5 (3)	7.1 ± 4.8	11.1–20.0	16.7 ± 4.9
Coriander (n = 3)	0.8–2.9 (3)	1.5 ± 1.1	7.5–18.0	13.2 ± 5.3	5.2–40.4 (3)	19.5 ± 18.5	5.7–12.7 (3)	8.5 ± 4.0	5.2–14.7	9.2 ± 4.9
Spinach (n = 3)	1.26–4.6 (3)	2.4 ± 1.9	19.6–22.5	21.2 ± 1.5	7.2–76.4 (3)	38.5 ± 23.2	2.0–15.8 (2)	9.3 ± 6.0	11.8–26.0	20.7 ± 7.7
Mirpurkhas										
Bitter gourd (n = 4)	0.58–1.7 (3)	1.3 ± 0.6	5.7–15.0	9.8 ± 7.8	2.7–11.1 (3)	6.7 ± 4.2	2.2–14.2 (3)	6.7 ± 6.1	9.5–21.5	16.7 ± 6.3
Cucumber (n = 4)	0.58–4.5 (3)	2.2 ± 2.0	20.2–33.4	25.5 ± 6.7	2.87–17.7 (3)	12.5 ± 8.3	11.6–14.8 (3)	12.7 ± 1.8	4.7–19.7	13.0 ± 7.7
Spinach (n = 3)	0.5–1.3 (3)	0.8 ± 0.4	10.0–21.5	14.2 ± 6.3	3.2–26.0 (3)	14.3 ± 8.6	1.2–15.8 (2)	8.7 ± 7.9	10.1–21.5	14.2 ± 6.3

* Total number of samples analyzed

[a] Number of samples above FAO/WHO (2001) recommended permissible limits

reasons for high soil metal concentrations that possibly restricted their movement into deeper horizons. Khan et al. (1994) recorded similar results in soils irrigated with city effluent at different villages near Faisalabad, Pakistan. Singh and Singh (1994), and Ghafoor et al. (1995) also reported similar metal concentrations and their pattern of distribution in soil profiles.

Comparison of metal concentrations with the safe limits of FAO/WHO (2001) showed that Cu and Ni concentrations were within the RPLin almost all the examined vegetables samples (Table 3). However, Cd, Pb, and Cr concentration were above the recommended permissible limits in most of the collected samples.

Cadmium concentrations in almost all the vegetables collected from all four sites were above the permissible limit (0.2 mg kg^{-1} dry weight) (Table 3). The highest mean Cd concentration (3.0 ± 4.7 mg kg^{-1}) was observed in cauliflower at Hyderabad which ranged from 0.1 to 8.4 mg kg^{-1} (dry weight) followed by spinach (2.8 ± 1.2 mg kg^{-1}) collected from Gujranwala which ranged from 0.9 to 4.3 mg kg^{-1}. The Cd values from the other two sites, Sialkot and Mirpurkhas were in the range of 0.3 mg kg^{-1} (gourd) to 2.90 mg kg^{-1} (coriander) and 0.50 mg kg^{-1} (spinach) to 4.50 mg kg^{-1} (cucumber), respectively. The mean Cd concentrations in all the vegetables were considerably lower than values (10.37–17.79 mg kg^{-1}) reported by Gupta et al. (2008) from Titagarh, India and by Turkdogan et al. (2002) from Turkey (25 mg kg^{-1}). However, the values were comparable with Cd concentrations of vegetables reported by Hussain et al. (2006) from Faisalabad, Pakistan (0.2–1.0 mg kg^{-1}) and by Sharma et al. (2007) from India (0.5–4.36 mg kg^{-1}), although they were higher than those reported by Dogheim et al. (2004) from Egypt (0.002–0.08 mg kg^{-1}) and by Liu et al. (2005) from China (0.03–0.73 mg kg^{-1}).

Similar to Cd, Pb concentrations in almost all the vegetables sampled were higher than the permissible limit of 0.3 mg kg^{-1} (Table 3). Vegetables collected from Gujranwala had the highest Pb concentration ranging from 0.1 mg kg^{-1} (chillies) to 90 mg kg^{-1} (spinach). Spinach samples from Gujranwala, Hyderabad and Mirpurkhas had higher Pb concentrations than in all other vegetables. The Pb concentration in spinach ranged from 15 to 90 mg kg^{-1} at Gujranwala, 7.2–76.4 mg kg^{-1} at Hyderabad and 3.2–26.0 mg kg^{-1} at Mirpurkhas. This is contrary to the studies of Pb mobility in leafy vegetables which have shown the partitioning of Pb is greatest in roots, followed by a decreasing trend in aboveground biomass (Finster et al. 2004). The elevated Pb concentrations in leafy vegetables may be a result of foliar intake of aerial Pb deposition attributed mainly to motor vehicles as Pb absorption through foliage is more pronounced at locations close to emission sources (Sharma and Prasad 2010). The mean Pb concentrations in all vegetables were several fold higher than the values reported in China [0.18–7.75 mg kg^{-1} by Liu et al. (2006)] and 1.97–3.81 mg kg^{-1} by Liu et al. (2005) as well as in India [3.09–15.74 mg kg^{-1} by Sharma et al. (2007)] but lower than the mean Pb concentration (409 mg kg^{-1}) reported in Turkey by Turkdogan et al. (2002).

The highest Cr concentrations (0.1–28.0 mg kg^{-1}) in vegetables were observed at Gujranwala followed by Sialkot (0.1–20.9 mg kg^{-1}), Hyderabad (0.2–15.8 mg kg^{-1}) and Mirpurkhas (1.2–15.8 mg kg^{-1}) (Table 3). The mean concentration of Cr in leafy vegetables was higher than that in other vegetables. For example, lettuce accumulated the highest Cr concentration (20.4 ± 2.4 mg kg^{-1}) followed by coriander (19.6 ± 7.4 mg kg^{-1}) and spinach (14.6 ± 6.5 mg kg^{-1}) in Gujranwala. In earlier studies (Hussain et al. 2006; Gupta et al. 2008) on vegetables grown in soils being irrigated with untreated municipal effluent around the Faisalabad, Pakistan and Titagarh, India the Cr concentrations measured were several times greater than those found in this study. More than 70% of the vegetable samples collected from Hyderabad and Mirpurkhas and more than 50% of those collected from Sialkot and Gujranwala had higher Cr concentrations than the permissible limit (2.3 mg kg^{-1}).

The study demonstrates the elevated concentrations of Cd, Pb and Cr in vegetables grown in soils irrigated with untreated effluent. Among the sampled vegetables, leafy vegetables showed the highest metal accumulations. These results suggest that the cultivation of leafy vegetables close to emission sources of Pb vapour should be avoided. Prolonged exposure to vegetables and fodder having elevated metal concentrations may cause serious health hazards for human and animals.

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