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Flux of Heavy Metals in Soils Irrigated with Urban Wastewaters

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Abstract: Heavy metals in waste waters, agricultural fields and vegetation are of major concern to human health. A study has been conducted to ascertain the addition of heavy metals into agricultural fields through city waste water irrigation. A *nalla* (water stream) running peripheral to Solapur (MS, India) city was a source for irrigation. The physicochemical analyses of nalla water and surrounding agricultural soils have revealed that the sites were heavily contaminated with heavy metals. The DTPA extractable metal concentrations in soils were found to be less than total metal content. Eleven vegetables, including leafy as well as tubers grown on fields irrigated with city waste water have been assessed for metal accumulation. Maximum mean metal accumulation values to the tune of 47.81, 31.35, 24.46, 42.92, 61.23 and 45.30 mg Kg^{-1} were obtained for copper in Green Sorrel (Chukka), chromium in Bitter gourd, cadmium in Spinach, lead in Spinach, zinc in Carrot and nickel in Radish, respectively. Leafy vegetable, Spinach appeared to be the highest metal accumulating plant on the basis of overall metal uptake. Among the six metals under study, zinc was most preferred metal in five plants followed by copper in four and lead in two plants. The results emphasized that there is an urgent need to study the effects of metal-accumulated vegetables on consumer society.

Key words: Urban wastewater · Metal accumulation · Phytoaccumulation · Metal flux

INTRODUCTION

Scarcity of surface/ground water for irrigation is an ever increasing problem around the world, owing to which, use of wastewater agriculture has become a common reality in three-fourth of the cities in Asia, Africa and Latin America [1]. Wastewater irrigation is practiced not only due to water scarcity but also due to nutritional value and environmental protection. A number of risk factors have been identified in wastewater reuse. Some of them are short termed and vary in severity (e.g., microbial pathogens), whereas others have longer-term impacts that increase with the continued use of recycled water (e.g., saline effects on soil) [2]. Presence of pollutants like heavy metals in urban and industrial waste waters results in contamination of water and soil. Household effluents, drainage water, business effluents, atmospheric deposition and traffic related emissions transported with storm water into the sewage system carry number of pollutants and enrich the urban waste water with heavy metals [3].

The characterization of soils receiving urban waste water is a necessity towards establishing the pollutional status. Metal transfer from biosolids to soil and subsequently to plants, pose potential health risks, since they can enter the food chain and the environment [4]. Mere increase in metal concentration of soil is not just sufficient for potential risk assessment. The total metal content of soil will usually include fractions that are not immediately available to plants, microorganisms and soil fauna [5]. Metal chemistry, speciation and thus bioavailability are known to be dependent on soil physicochemical parameters. The mobility of trace metals, their bioavailability and related eco-toxicity to plants, depend strongly on their specific chemical forms or ways of binding [4]. However, many studies have demonstrated good correlation between total metal content and uptake by plants [6]. Strong binding of heavy metals to the soil particles reduces the bioavailability resulting in less plant uptake [7]. Soil erosion and plant uptake are the ways for loss of metals from soil. Still, heavy metals are considered to be less mobile and thus less bioavailable in soils rich in organic matter and clay contents [7].

Uptake of heavy metals in plants is of major concern as it may reach humans through food web [1]. Plants can accumulate higher amounts of metals if they are grown in

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polluted soils. Prolonged exposure to heavy metals such as cadmium, copper, lead, nickel and zinc can cause deleterious health effects in humans [8].

Solapur is a city (17.68° N 75.92°E) located in southern parts of Maharashtra state in India. The climate of Solapur is dry. Summer, monsoon and winter are the three seasons of the city. The maximum temperature in summer season ranges from 30 to 40°C. The average annual rainfall is 545 mm. The city waste waters runs through a peripheral water stream (*nalla*) around the city. This water has been lifted directly for agriculture purpose. This waste water stream and agricultural fields irrigated with these waste waters was the study area. Vegetables and other cash crops have been cultivated using this water.

In the present study we have investigated the heavy metal content of urban waste waters, soils and vegetables under cultivation. The objectives of this work were to establish the pollutional status of sites and study the flux of heavy metals from waste waters into agricultural soils and further entering into food web.

MATERIALS AND METHODS

Sample Collection: Waste water samples were collected from four different locations along the waste water stream (*nalla*) running around the Solapur city. Water samples were collected in sterile plastic bags and carried to the laboratory. Soil samples were collected from the agricultural fields near the waste water stream covering a distance of 3 Km. Soil sampling was conducted four times in a year beginning from January 2005 to December 2007. In total 16 soil samples were collected from each site. All soil samples were taken from the surface layer (0-25 cm) of cultivated soils. After thorough mixing, 200 g representative samples from each field were stored in polythene bags. The soils were air dried, homogenized with mortar and pestle and sieved through 1 mm mesh. Water and soil samples were analyzed in the laboratory as per the standard procedures [9-11].

Eleven vegetables viz. Onion (*Allium cepa* L.), Coriander (*Coriander sativum* L.), Cucumbar (*Cucumis sativus* L.), Carrot (*Daucus carota* L.), Tomato (*Lycopersicon esculentum* Mill.), Bitter gourd (*Memordica charantia),* Radish (*Raphanus sativus* L.), Green Sorrel (*Rumex vesicarius* L.), Brinjal (*Solanum melongena),* Spinach (*Spinacia oleracea* L.) and Methi (*Trigonela foenum* L.) were collected randomly in triplicate from fields irrigated with waste waters during 2005 to 2007. The vegetables were washed thoroughly with distilled water for the removal of soil particles. Vegetable were sun dried, crushed and ground to powder used for metal analysis.

Digestion Method: In order to estimate the total metal content, soil and dried vegetable samples were digested with aqua-regia (3:1, hydrochloric acid + nitric acid) at 150°C for 2 h in the COD digester (Model: 2015M, Make: Spectralab Inst. Pvt Ltd., India). The digested samples were cooled and filtered through Whatman No.1 filter paper and then the volumes were made up to 50 ml using volumetric flasks.

Metal Extraction: To estimate the available metal concentration, 10 g dried soil was mixed with 20 ml of diethylenetriamine pentaacetic acid (DTPA) extracting solution at pH 7.3 and kept on rotary shaker at 120 rpm for 2 h. The mixture was centrifuged at 5000 rpm for 5 min and supernatants were collected and acidified with 1:1 nitric acid for metal analysis.

Metal Analysis: Metal analysis of all digested samples and acidified wastewaters was carried out by atomic absorption spectrometry (Model: S_2, Make: Thermo, USA) using the 'SOLAAR' software. Analyses were performed using hollow cathode lamps for copper (Cu), chromium (Cr), cadmium (Cd), lead (Pb), zinc (Zn) and nickel (Ni) at 324.8, 357.9, 228.8, 217.0, 213.9 and 232.0 nm, respectively. Air-acetylene flame was generated using a fuel flow rate of 0.8 to 1.1 L min^{-1}. All analyses were replicated three times. All the reagents used were analytical grade (Thermo Fischer Sci. India Pvt. Ltd. and HiMedia Lab. Pvt. Ltd., India).

RESULT AND DISCUSSION

Physiochemical Properties of Nala Water and Soils: The physicochemical analysis of *nalla* water receiving city waste waters is presented in Table 1. The results show that metal composition of *nalla* water was complex and highly variable. It could be seen that pH of *nalla* water at four different sites ranged between 6.98 and 7.81. These values satisfy the pH necessary for irrigation (6.0 – 9.0) [12]. Electrical conductivity of *nalla* water has ranged from 0.90 to 1.98 dS m^{-1}. EC values <2 have indicated less salt contents which will not add salts to the soils [13]. Heavy metal analysis of *nalla* water was done to check the suitability for agricultural use. Heavy metal concentration (mg L^{-1}) in *nalla* waters at different locations ranged from 5.23 to 11.28 for copper, 13.02 to 35.18 for chromium, 4.54 to 25.38 for cadmium, 5.32 to 11.13 for lead, 8.54 to 22.23 for zinc and 4.98 to 18.32 for nickel.

Table 1: Physicochemical analysis of *nalla* water running peripheral to Solapur city

	Samples collected in three years 2005, 2006 and 2007			
Parameter	Jan-Mar	Apr-June	July-Sept	Oct-Dec
pH	7.28 (0.58)^{a}	7.38 (0.81)	7.81 (0.45)	6.98 (0.81)
EC (dS m^{-1})	1.58 (0.42)	1.98 (0.51)	0.90 (0.44)	1.54 (0.28)
Cu (mg L^{-1})	9.75 (2.24)	7.54 (1.82)	5.23 (1.05)	11.28 (2.88)
Cr (mg L^{-1})	35.18 (6.12)	24.23 (5.98)	13.02 (6.32)	18.38 (8.44)
Cd (mg L^{-1})	25.38 (6.92)	18.22 (5.19)	4.54 (4.24)	16.32 (8.12)
Pb (mg L^{-1})	9.23 (5.83)	11.13 (6.88)	5.32 (6.24)	8.22 (5.09)
Zn (mg L^{-1})	18.56 (8.43)	21.01 (6.44)	8.54 (7.32)	22.23 (6.18)
Ni (mg L^{-1})	9.43 (6.05)	18.32 (8.22)	4.98 (4.72)	7.54 (6.42)

All values are mean values; a, Figures in parentheses are of standard deviation (n= 12 each quarter)

The concentrations of heavy metals were in the order of: Pb < Cu < Ni < Zn < Cd < Cr. The concentrations of heavy metals were found to be far higher for irrigation purpose when compared with the permissible concentration of heavy metals in waste waters [1, 14]. The higher standard deviations in all parameters analyses for the samples of different locations and collected at different time periods could be attributed to the diversified anthropogenic activities. Decreased values of EC and metal concentrations in the samples collected during July-September might be owing to dilution of *nalla* water with rain water. Increase in EC during April-June was due to concentration of *nalla* water in summer as the increased temperature results in more evaporation and scarcity of water.

Presence of high metal concentrations in *nalla* water may deteriorate the agricultural soils and also poses health hazards to humans. As there is an increased risk of heavy metals flux into food web through agricultural cultivates. However, the advantages that farmers found are that they could raise vegetables in summer when there is a great demand in market and they need not wait for rains unlike in the rainfed situation [15].

The soil physicochemical properties have great influence on availability of nutrients and metals [16]. The solubility of metallic ions depends on various soil physiochemical factors, such as the pH, type and density of the charge on soil colloids and the reactive surface area [17]. The major factors governing the availability of metallic ions to plants are the solubility and the thermodynamic activity of the uncomplexed ion [18].

In the present study, analysis of soils receiving *nalla* water was performed to study the effect of soil properties on availability and thus the mobility of metals. The data of soil analysis are presented in Table 2. Soils of sampling sites belonged to loam, silt and sludge types. It can be seen that silt and sludge soils showed acidic pH against alkaline pH of loamy soils. Soil pH gets affected to a certain extent due the application of various amendments. Increase in soil pH increases immobilization of heavy metals through the process of ion exchanges and surface complexation [19].

Electrical conductivity of soils was <1.5 with the exception of Site-4 having loamy soil. Total organic carbon is another important parameter plays a major role in nutrient cycling and also indicates the quality of soil. Increase in total organic carbon could be attributed to the addition of municipal waste water [20]. High water holding capacity ranging between 44.60 to 73.26% was might be due to the fine soil structures. Sodium adsorption ratios (SAR) were in the range of 3.41 to 6.68. Rise in SAR signified that there was no risk of sodium hazard from irrigation with *nalla* water. Changes in water holding capacity, SAR, total organic carbon could be attributed to continuous addition of waste water.

Total and Extractable Metal Contents of Soil: Results of heavy metal analysis performed for the soils irrigated with *nalla* water are presented in Table 3. Among the six metals tested, lead, copper and zinc concentrations were higher than chromium, cadmium and nickel. High metal concentrations in soil are one of the important environmental concerns [3]. The use of total concentration as a criterion to assess potential effects of soil contamination is not sufficient, because fate and toxicity of heavy metals in a contaminated soil is greatly controlled speciation in the soil [21]. Total metal concentrations may not be the best predictor of metal bioavailability in soils and therefore study on bioavailability is becoming more important in ecological risk assessments [22]. Heavy metal contents in plants can be predicted easily for elements, which are bounded with low binding strength to the soil [23]. In order to determine the bioavailability and mobility of metals,

Table 2: Physicochemical analysis of soil receiving the *nalla* water

Parameter \ Site	Site-1	Site-2	Site-3	Site-4
Soil texture	Lome	Silt	Sludge	Lome
pH	7.8 (0.89)[a]	6.6 (0.66)	5.8 (0.57)	7.5 (0.83)
EC (S m^{-1})	0.72 (0.65)	0.92 (0.58)	1.14 (1.12)	2.24 (1.72)
$CaCO_3$ (%)	2.14 (1.65)	1.93 (0.94)	1.45 (1.06)	2.22 (1.84)
Total organic carbon, mg L^{-1}	28.18 (7.21)	42.32 (8.54)	55.14 (6.77)	24.97 (11.14)
Water holding capacity (%)	44.60 (12.06)	58.12 (10.22)	62.55 (14.14)	73.26 (8.96)
CEC, mEq $100g^{-1}$	16.41 (3.75)	27.18 (4.52)	13.23 (4.66)	23.96 (2.76)
SAR	5.66 (1.65)	5.24 (1.80)	3.41 (0.94)	6.68 (1.38)

All values are mean values; a, Figures in parentheses are of standard deviation (n= 16 at each site)

Table 3: Total and extractable metals in soils irrigated with city wastewaters

	Metal concentration (mg Kg^{-1})							
	Site-1		Site-2		Site-3		Site-4	
Metal	Tot	Ext	Tot	Ext	Tot	Ext	Tot	Ext
Cu	155.11	67.04	178.12	60.24	209.03	89.63	211.08	89.53
	(22.16)[a]	(16.16) [43.22][b]	(25.08)	(18.43) [33.82]	(12.61)	(18.13) [42.88]	(23.44)	(19.53) [42.42]
Cr	57.56	37.32	73.66	46.88	97.75	56.98	47.97	29.66
	(8.92)	(14.06) [64.84]	(12.36)	(5.84) [63.64]	(7.18)	(5.68) [58.29]	(14.06)	(7.78) [61.83]
Cd	85.27	34.01	75.23	24.75	65.26	23.86	115.27	48.55
	(13.15)	(4.92) [39.89]	(17.03)	(4.55) [32.90]	(13.40)	(4.02) [36.56]	(16.86)	(6.66) [42.12]
Pb	133.32	55.35	129.16	66.82	84.77	31.62	134.19	43.08
	(14.46)	(7.12) [41.52]	(22.46)	(7.42) [51.73]	(5.74)	(4.44) [37.30]	(13.04)	(9.11) [32.10]
Zn	231.27	74.45	214.66	148.76	189.53	113.98	165.73	79.45
	(19.88)	(11.55) [32.19]	(15.02)	(8.30) [69.30]	(11.18)	(7.04) [60.14]	(14.14)	(6.70) [47.94]
Ni	54.03	26.45	48.88	28.23	51.66	26.77	80.76	37.87
	(11.05)	(6.73) [48.95]	(6.23)	(3.55) [57.75]	(8.15)	(3.89) [51.82]	(7.46)	(4.88) [46.89]

All values are mean values; Tot, Total metal concentration; Ext, Metal concentration extracted with DTPA; a, Figures in parentheses are of standard deviation (n= 16 at each site); b, Figures in rectangle bracket are of % extraction efficiency

the soil samples were extracted with DTPA. The DTPA extractable metal concentrations are given in Table 3. Extractable metal in soil samples were lower as compared to the total content of metals. Maximum 64.84% extraction was seen in case of chromium. Low % extraction of other metals has indicated the formation of high affinity complexes of metal and soil particles. Elevated levels of heavy metals in soils could be ascribed to prolonged use of *nalla* water for irrigation.

A variety of extracting agents have been used to assess the bioavailable metal concentrations in the soil [4, 13, 22, 23]. The % metal extraction and speciation of metals is dependent on pH, EC, $CaCO_3$, organic carbon contents, type of soil and method of extraction. EDTA solution is assumed to extract principally organically bound and carbonate bound fractions of metal by forming strong soluble complexes [4]. There is no fully satisfactory extractant for all soil-plant systems because of varying properties of different soils and plant species [19]. However, EDTA is more suitable for acidic soils but not for neutral or alkaline soils, whereas, DTPA was considered more suitable for neutral and near alkaline soils, as it buffered at pH 7.3 and therefore prevents $CaCO_3$ from dissolution and release of occluded metals [19, 22]. In present study, the use of DTPA for metal extraction from neutral-alkaline soils has given an approximation to the extent of effective metal concentration that would be entering the food web. Malla *et al.* [24] have reported an improvement in the fertility status of the soils but with build up of metallic cations in the soil upon sewage water irrigation.

Metal Accumulation in Vegetables: Use of untreated effluents or *nalla* waters in agricultural soils may give rise to accumulation of heavy metals in the top soil and hence in the vegetation. In order to check the flux of heavy metals, vegetables grown on waste water were analyzed for metal contents.

Table 4: Accumulation of heavy metals in vegetables grown in *nalla* water irrigated soils

		Metal concentration (mg Kg^{-1} dry weight)					
Vegetable	Family	Cu	Cr	Cd	Pb	Zn	Ni
Allium cepa L. (Onion)[a] (n= 52)[b]	*Liliaceae*	41.68 (14.44)[c]	11.82 (5.44)	4.10 (1.97)	11.30 (2.98)	28.32 (7.34)	8.56 (4.04)
Coriander sativum L. (Coriander) (n= 56)	*Apiaceae*	31.42 (18.03)	17.23 (6.21)	14.99 (3.98)	20.46 (6.24)	23.22 (8.01)	13.64 (4.35)
Cucumis sativus L. (Cucumbar) (n= 48)	*Cucurbitaceae*	5.56 (5.10)	12.35 (4.34)	4.54 (2.18)	41.43 (8.37)	12.60 (4.83)	18.43 (6.26)
Daucus carota L. (Carrot) (n= 42)	*Brassicaeae*	27.12 (10.88)	8.38 (2.67)	7.85 (4.02)	8.97 (2.84)	61.23 (12.07)	10.32 (4.06)
Lycopersicon esculentum Mill. (Tomato) (n= 48)	*Solanaceae*	27.75 (9.32)	20.32 (7.13)	10.17 (4.45)	5.23 (2.07)	42.13 (8.37)	34.88 (13.11)
Memordica charantia L. (Bitter gourd) (n= 52)	*Cucurbitaceae*	42.12 (8.14)	31.35 (9.16)	9.58 (3.08)	4.25 (2.83)	18.23 (6.06)	27.5 (9.12)
Raphanus sativus L. (Radish) (n= 46)	*Brassicaceae*	36.32 (12.10)	21.21 (7.08)	16.18 (4.96)	8.47 (3.14)	52.98 (14.18)	45.3 (13.04)
Rumex vesicarius L. (Green Sorrel) (n= 52)	*Polygonaceae*	47.81 (18.08)	12.83 (4.18)	7.94 (3.27)	2.73 (1.82)	22.54 (6.71)	13.8 (4.62)
Solanum melongena L. (Brinjal) (n= 56)	*Solanaceae*	9.54 (6.21)	12.34 (3.93)	5.48 (2.88)	4.64 (2.34)	32.52 (8.03)	7.9 (3.81)
Spinacia oleracea L. (Spinach) (n= 42)	*Polygonaceae*	32.95 (16.32)	18.31 (5.45)	24.46 (6.41)	42.92 (8.41)	18.71 (4.04)	36.62 (13.06)
Trigonela foenum L. (Methi) (n= 42)	*Fabaceae*	8.50 (5.94)	9.08 (3.06)	19.86 (4.55)	6.77 (3.52)	40.62 (7.73)	10.77 (4.09)

All values are mean values; a, Vernacular names of vegetables; b, Number of vegetable samples collected in three years; c, Figures in parentheses are of standard deviation

Table 5: Order of metal accumulation in vegetables grown in *nalla* water irrigated soils

Vegetable	Order of metal accumulation
Allium cepa L. (Onion)	Cu > Zn > Cr > Pb > Ni > Cd
Coriander sativum L. (Coriander)	Cu > Zn > Pb > Cr > Cd > Ni
Cucumis sativus L. (Cucumbar)	Pb > > Ni > Zn > Cr > Cu > Cd
Daucus carota L. (Carrot)	Zn > > Cu > Ni > Pb > Cr > Cd
Lycopersicon esculentum Mill. (Tomato)	Zn > Ni > Cu > Cr > Cd > Pb
Memordica charantia L. (Bitter gourd)	Cu > Cr > Ni > Zn > Cd > Pb
Raphanus sativus L. (Radish)	Zn > Ni > Cu > Cr > Cd > Pb
Rumex vesicarius L. (Green Sorrel)	Cu > > Zn > Ni > Cr > Cd > Pb
Solanum melongena L. (Brinjal)	Zn > Cr > Cu > Ni > Cd > Pb
Spinacia oleracea L. (Spinach)	Pb > Ni > Cu > Cd > Zn > Cr
Trigonela foenum L. (Methi)	Zn > > Cd > Ni > Cr > Cu > Pb

Table 4 gives the distribution of six metals in vegetables collected from various sites in different seasons for three years. Maximum mean metal accumulation values to the tune of 47.81, 31.35, 24.46, 42.92, 61.23 and 45.30 mg Kg^{-1} were obtained for Cu (Green Sorrel), Cr (Bitter gourd), Cd (Spinach), Pb (Spinach), Zn (Carrot) and Ni (Radish), respectively. Leafy vegetable, Spinach appeared to be the highest metal accumulating plant on the basis of overall metal uptake. Higher standard deviations in metal accumulation values could be due to breed variety of vegetables as well as soil types and seasonal variations. Order of preference to the metals for vegetables prepared on the basis of metal accumulation values are listed in Table 5. Among the six metals under study, zinc was the most preferred metal in five plants followed by copper in four and lead in two plants. Spinach and cucumber emerged as potential accumulators for lead. Lead and cadmium were found to be the least preferred metals for six and three vegetables.

The results obtained in present study are in agreement with previous studies showing increased levels of heavy metals in edible food crops grown in wastewater irrigated soils [1, 25, 26, 27]. These reports demonstrate that the plants grown on waste-water irrigated soils are generally contaminated with heavy metals, posing a major health concern [25]. Arora *et al.* [25] have reported accumulation of metals in the range of 116-378, 12-69, 5.2-16.8 and 22-46 mg Kg^{-1} for iron, manganese, copper and zinc, respectively. Further, highest accumulation of iron and manganese were obtained with mint and spinach, whereas the level of copper and zinc were highest in carrot. The variation in the per cent build up of metallic cations in the soils and vegetable crops may be attributed to a greater variation in the initial values of the metallic cations in the soil prior to experimentation and preferential absorbance of a particular cation by different vegetable crops under study [24].

Besides, many studies and reports confirming the flux of heavy metals from wastewater irrigated soils to edible vegetables prove that the practice is still continued due to one or other reasons. The advantages that farmers found are that they could raise vegetables in summer when there is a great demand in the market and they need not wait for rains unlike in the rainfed situation [15].

CONCLUSIONS

The scarcity of water forces mankind to use alternative, easily available and cheaper water sources. Farmers are attracted to use *nalla* water, a water stream carrying composite liquid waste from domestic and industrial activities in the city for irrigation purpose. The present study was an attempt to establish the mobility of toxic heavy metals from *nalla* water into food web via metal-accumulating vegetables. The physicochemical analysis of *nalla* water and soil samples have indicated heavy load of metals resulting into continuous addition to the agricultural fields. Differences in total and DTPA extracted metal concentration have suggested immobilization of heavy metals into soil by forming high affinity complexes. However, the eleven vegetables collected from the region grown on *nalla* water have shown increased metal accumulation. Presence of heavy metals in vegetables poses serious health hazards. The results of the present study necessitate further work on tracking down the mobility of heavy metals along the food web.

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