

HEAVY METALS CONTAMINATION OF SOILS IN RESPONSE TO WASTEWATER IRRIGATION IN RAWALPINDI REGION

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The study was conducted to evaluate the quality of effluents/ waste water samples from Rawalpindi region for irrigation purpose and to elucidate effects of their application on heavy metal contents in soils of area. Results indicated that the EC, SAR, RSC and TDS of most effluent/ waste water samples were above the critical limits. Cadmium and Cr were above the critical limits in almost all the effluent samples, whereas Ni was high in 14, Pb was high in 10, Cu was high in 5 and the Fe was high in 3 effluent samples as compared to critical limits. Regarding heavy metals contents of soils irrigated by these effluents/ waste water, total Fe, total Cd and total Ni were higher in almost all the sampled sites, whereas total Cr was high at 7 sampled sites. AB-DTPA extractable Fe and Zn were higher at all the sampled sites, while the extractable Cd was higher at 2 sampled sites. Overall, the effluent samples collected from Adiala showed high concentrations of heavy metals, whereas soils of Wah factory and Islamabad area had higher heavy metal contents (total & AB-DTPA extractable). On the basis of results it is concluded that quality of effluents/ waste water samples collected from different locations of Rawalpindi is not good for irrigation and the long term use of these effluents for crop production caused accumulation of some toxic metals in soils above critical limits which is harmful for soil health and may lead to elevated levels of heavy metals in crop plants.

Keywords: Wastewater, AB-DTPA extractable and total heavy metals, physico-chemical parameters

INTRODUCTION

Application of domestic and industrial effluents to nearby agricultural fields is a common practice of irrigation and wastewater disposal in Pakistan (Lone *et al.*, 2000; Ensink *et al.*, 2004). Saline groundwater, a poorly performing irrigation system, and recurrent droughts have led to increased water shortages due to which use of untreated wastewater for agriculture has become a common and widespread practice. Besides providing supplemental irrigation, wastewater is a useful source of plant nutrients, particularly the nitrogen and phosphorous and of organic matter to improve soil fertility and physical properties (Gibbs *et al.*, 2006). However, besides these beneficial effects wastewater often contains appreciable amounts of organic and inorganic toxic materials. The organic pollutants being biodegradable are less persistent, and presumably have transient and less serious effects in soil environment as they eventually metabolize to carbon dioxide and other simpler products. Among the inorganic substances, heavy metals are often present in substantial quantities chelated by the organic matter in wastewater. When wastewater is applied to agricultural fields, heavy metals enter the soil and get fixed to the soil components. Thus continuous

application of wastewater tends to accumulate large quantities of heavy metals in soil, which persists there for an indefinite period to have long lasting effects in the soil environment (Kabata-Pendias and Pendias, 2002).

Inhibition of root growth, shoot development and various metabolic processes in plants have been reported because of higher concentrations of heavy metals in soils which further resulted in chlorosis, damage to root tips, reduced water and nutrient uptake and damage to enzyme system (Baisberg-Pahlsson, 1989; Sanita di Toppi and Gabbrielli, 1999). Chronic lower level intakes of toxic elements have damaging effects on human beings and other animals (Ikeda *et al.*, 2000), since there is no efficient mechanism for their elimination, and the detrimental impact becomes apparent only after several years of exposure (Bahemuka and Mubofu, 1999). Consuming food contaminated by Pb, Hg, As, Cd and other metals can seriously deplete body stores of Fe, vitamin C and other essential nutrients, leading to decreased immunological defences, intrauterine growth retardation, impaired psycho-social faculties and disabilities associated with malnutrition (Iyengar and Nair, 2000). Thus, it is extremely important to monitor heavy metal contents in waste water irrigated soil in

order to prevent the entry of heavy metals into food chain. At present, there is little published information on the extent of heavy metals contamination of soils by effluents/ waste water irrigation particularly in Rawalpindi region of Pakistan. The objective of present study was to quantify the contents of heavy metals in effluents/ wastewater samples collected from different areas of Rawalpindi and to evaluate the extent of soil contamination by heavy metals in response to effluents/ waste water irrigation.

MATERIALS AND METHODS

Twenty three farmers fields *i.e.*, 5 from Adiala, 8 from Pirwadai/ Islamabad area, 5 from Taxilla and 5 at Wah Factory sites having been irrigated with effluents/ wastewater for more than 10 years were selected for the collection of soil and water samples during the year 2007-08. Effluent/ wastewater samples were taken at a depth of 15 cm using a measuring cylinder, one drop of HCl was added and stored in pre-rinsed plastic bottles. The samples were analyzed within 20 days after their collection for pH, electrical conductivity (EC), sodium adsorption ratio (SAR), residual sodium carbonate (RSC) and total dissolved solids (TDS) by the methods described in USDA Agricultural Handbook No 60 (US Salinity Laboratory Staff, 1954). Soil samples were collected at 0-15 cm depths from each of the selected sites. The samples were air-dried, ground, passed through a 2 mm sieve, mixed thoroughly and analyzed for the selected soil physical and chemical properties such as particle size analysis, EC, pH, CEC, CaCO_3 , organic C, and total and AB-DTPA extractable heavy metals. Particle size analysis was carried out by the hydrometer method (Bouyoucos, 1962). EC, pH, CEC and CaCO_3 were determined by the methods given in the USDA Agricultural Handbook No 60 (US Salinity Laboratory Staff, 1954). Ammonium bicarbonate-diethylene triamine pentaacetic acid (AB-DTPA) extractable heavy metals (Cu, Zn, Cd, Pb, Ni, Cr, Fe) were determined by the method of Soltanpour (1985). Ten gram soil was extracted with 20 ml of AB-DTPA after 15 minutes shaking at 180 rev min^{-1} followed by filtration through Whatman No. 42 filter paper and the metals were determined using atomic absorption spectrophotometer (AAS) GBC-932 Plus, UK. The instrument (AAS) was calibrated with a series of standard solutions supplied by the manufacturer (Varian). Total heavy metals (Cu, Zn, Cd, Pb, Ni, Cr, Fe) in the soil samples were estimated after *aqua regia* digestion (Soon and Abboud, 1993). Organic C was estimated by the method described by Page *et al.* (1982). All the soil and water estimations were

performed in triplicate along with blanks to minimize error. The data were subjected to statistical analysis such as the mean values, standard deviation (SD) and correlation using MS-Excel software.

RESULTS

Chemical Properties and Heavy Metal Concentration in Waste Water:

Electrical conductivity (EC) of the waste water samples from all 23 sites ranged from 0.6 to 2.8 dSm^{-1} with a mean value of 1.4 dSm^{-1} (Table 1). The samples from Islamabad had the highest (2.0 dSm^{-1}) mean EC, whereas the samples from Taxilla had the lowest (0.9 dSm^{-1}). The pH of the waste water samples ranged from 6.8 to 8.6 with a mean value of 7.7. The maximum mean pH (8.1) was observed in Adiala samples, while the minimum (8.1) in Wah factory samples. The total dissolved solids (TDS) in waste water samples were in the range of 436 to 1993 mg L^{-1} with a mean value of 980 mg L^{-1} . Mean maximum (1417 mg L^{-1}) TDS value was observed at Islamabad, whereas the minimum (648 mg L^{-1}) at Taxilla site. The sodium adsorption ratio (SAR) of the waste water samples ranged from 0.4 to $4.8 \text{ mmol}^{1/2} \text{ L}^{-1/2}$ with the mean value of $2.4 \text{ mmol}^{1/2} \text{ L}^{-1/2}$. The maximum mean SAR ($3.6 \text{ mmol}^{1/2} \text{ L}^{-1/2}$) was observed at Islamabad while the minimum ($1.3 \text{ mmol}^{1/2} \text{ L}^{-1/2}$) at the Adiala site. The residual sodium carbonate (RSC) ranged from 0.1 to $2.0 \text{ mmol}_c \text{ L}^{-1}$ at all the sampled sites with a mean value of $0.4 \text{ mmol}_c \text{ L}^{-1}$. The highest mean RSC value ($0.9 \text{ mmol}_c \text{ L}^{-1}$) was observed at Adiala, whereas the minimum ($0.1 \text{ mmol}_c \text{ L}^{-1}$) at the Islamabad site.

Zinc concentration in the waste water samples ranged from 0.01 to $0.80 \text{ } \mu\text{g mL}^{-1}$ with a mean value of $0.09 \text{ } \mu\text{g mL}^{-1}$ (Table 1). The waste water samples from Wah factory area showed the higher mean concentration of Zn ($0.22 \text{ } \mu\text{g mL}^{-1}$), whereas the samples from Taxilla had the minimum ($0.01 \text{ } \mu\text{g mL}^{-1}$). The Cd ranged from 0.0 to $0.13 \text{ } \mu\text{g mL}^{-1}$ with a mean value of $0.03 \text{ } \mu\text{g mL}^{-1}$ at all the sampled sites. The samples from Wah factory showed the higher mean concentration of Cd ($0.05 \text{ } \mu\text{g mL}^{-1}$) whereas the minimum mean concentrations were observed at Taxilla ($0.02 \text{ } \mu\text{g mL}^{-1}$) and Islamabad sites ($0.02 \text{ } \mu\text{g mL}^{-1}$). The Fe concentration ranged from 0.03 to $9.14 \text{ } \mu\text{g mL}^{-1}$ with a mean value of $1.30 \text{ } \mu\text{g mL}^{-1}$. The samples from Adiala showed the higher mean Fe concentration ($2.95 \text{ } \mu\text{g mL}^{-1}$), whereas the Islamabad samples had the minimum ($0.22 \text{ } \mu\text{g mL}^{-1}$). The concentration of Cu ranged from 0.1 to $0.45 \text{ } \mu\text{g mL}^{-1}$ with a mean value of $0.10 \text{ } \mu\text{g mL}^{-1}$. The higher mean Cu concentration ($0.22 \text{ } \mu\text{g mL}^{-1}$) was observed in wastewater samples from Adiala, whereas the

Table 1. Chemical properties and heavy metals concentration of wastewater samples

Sampling sites	EC dS m ⁻¹	pH	TDS mg l ⁻¹	SAR (m mol ⁻¹) ^{1/2}	RSC meq l ⁻¹	Zn µg ml ⁻¹	Fe µg ml ⁻¹	Cu µg ml ⁻¹	Cd µg ml ⁻¹	Cr µg ml ⁻¹	Pb µg ml ⁻¹	Ni µg ml ⁻¹
1) Adiala	0.8-	7.9-	562-	0.4-2.3	0.0	0.01-	0.31-	0.14-	0.02-	0.02-	0.08-	0.00-
(Range)	1.7	8.6	1183			0.35	9.12	0.45	0.05	0.75	0.24	0.54
Mean	1.1	8.1	784	1.5	0.0	0.14	2.95	0.22	0.03	0.27	0.13	0.27
CV±(%)	6.1	0.8	1.4	2.9	0.0	12.8	9.2	13.5	13.5	11.2	10.3	5.9
2) Pirwadhai	0.9-	7.1-	640-	1.8-4.8	0.0-	0.00-	0.04-	0.00-	0.01-	0.01-	0.00-	0.13-
(Range)	2.8	8.1	1993		2.0	0.17	0.34	0.04	0.03	0.25	0.08	0.56
Mean	2.0	7.8	1417	3.6	0.9	0.04	0.22	0.02	0.02	0.13	0.03	0.26
CV±(%)	3.5	0.2	0.9	2.3	10.1	17.1	9.8	12.2	17.1	11.7	12.3	10.0
3) Taxilla	0.6-	7.0-	436-	0.9-3.1	0.0-	0.00-	0.10-	0.00-	0.00-	0.03-	0.00-	0.02-
(Range)	1.7	8.1	1180		0.5	0.02	1.40	0.10	0.04	0.15	0.12	0.40
Mean	0.9	7.6	648	1.9	0.1	0.01	0.51	0.02	0.02	0.09	0.03	0.18
CV±(%)	2.4	0.2	1.6	2.9	3.1	15.6	4.5	8.3	7.5	11.5	8.9	7.4
4) Wah factory	0.8-	6.8-	571-	1.5-4.0	0.1-	0.00-	0.03-	0.01-	0.00-	0.09-	0.00-	0.01-
(Range)	1.8	7.5	1300		1.4	0.80	6.54	0.30	0.13	0.34	0.14	0.40
Mean	1.2	7.1	776	2.4	0.20	0.22	1.72	0.15	0.05	0.19	0.05	0.22
CV±(%)	3.2	1.0	1.6	3.7	2.5	11.6	8.7	9.3	10.1	8.2	8.9	14.1
* Permissible limit (FAO)	0.7	6.5-8.4	< 450	3.0	2.0	2.0	5.0	0.2	0.01	0.1	5.0	0.2
**Permissible limit (WWF)	1.5	6.5-8.4	1000	8	1.25	2.0	5.0	0.2	0.01	0.01	0.1	0.2

Critical limits as described by FAO, (1985), ** Critical limits as described by WWF, (2007) for Pakistan.

minimum (0.15 µg ml⁻¹) at Wah factory site. The Cr and Ni concentrations in wastewater ranged from 0.01 to 0.75 µg ml⁻¹ and from 0.00 to 0.56 µg ml⁻¹ with mean values of 0.18 and 0.25 µg ml⁻¹, respectively. The highest mean Cr concentration (0.27 µg ml⁻¹) was observed in Adiala site samples while the minimum (0.09 µg ml⁻¹) at the Taxilla site. Nickel showed the same trend like Cr with maximum mean value of 0.27 µg ml⁻¹ at Adiala and the minimum (0.18 µg ml⁻¹) at Taxilla site. The Pb concentration ranged from 0.00 to 0.24 µg ml⁻¹ with a mean value of 0.06 µg ml⁻¹. Water samples from Adiala had the higher mean concentration of Pb (0.13 µg ml⁻¹), whereas the Taxilla and Islamabad sites had the minimum (0.03 µg ml⁻¹). Overall, the total heavy metals contents in wastewater samples were in the order: Adiala > Wah factory > Islamabad > Taxilla.

Physical and Chemical Properties of Soils Irrigated with Waste Water

EC of soil samples ranged from 0.25 to 1.15 dSm⁻¹ with a mean value of 0.60 dSm⁻¹ (Table 2). Maximum mean EC value was observed in Islamabad (0.70 dSm⁻¹), whereas minimum in Wah factory (0.47 dSm⁻¹) soils. Soil pH_s value ranged from 7.4 to 8.1 with mean values of 7.8. Mean maximum pH value was observed in Islamabad (7.9), whereas the minimum in Wah factory (7.6) soils. Percent CaCO₃ contents ranged from 2.9 to 21.3 % with a mean value of 12.9 %, in all

sampled sites (Table 2). High mean CaCO₃ (%) values was observed at Adiala (14.4 %) and the minimum at Islamabad site (10.9 %). Organic C ranged from 0.32 to 1.09 % with a mean value of 0.7 % (Table 2). The organic C contents showed higher values at Taxilla site (0.80 %) and the minimum at Wah factory (0.50 %). Cation exchange capacity (CEC) ranged from 12.4 to 25.4 C mol_c kg⁻¹ with a mean value of 18.6 C mol_c kg⁻¹ (Table 2). High mean CEC values were observed at Islamabad (20.4 C mol_c kg⁻¹) and lowest at Adiala (17.2 C mol_c kg⁻¹) sites. Soil texture at Islamabad sites ranged from loam to clay loam, at Adiala ranged from sandy loam to sandy clay loam, at Taxilla sites ranged from loam to sandy loam and at Wah factory from loam to clay loam. Out of 23 sampled sites, 8 samples belonged to loam, 6 to sandy clay loam, 4 to sandy loam, 4 to clay loam and 1 to clayey soil textural classes.

Heavy Metal Contents of Soils Irrigated with Waste Water

Total heavy metals: Data of total zinc accumulation in soil ranged from 7.7 to 200 µg g⁻¹ with mean value of 49.7 µg g⁻¹ (Table 3). Maximum mean total Zn concentration (69.7 µg g⁻¹) was observed in Islamabad soil samples whereas minimum mean total Zn value was seen in Taxilla (20.5 µg g⁻¹) samples. Total Cd concentration ranged from 0.4 to 3.5 µg g⁻¹ with average mean values of 2.0 µg g⁻¹ at all sampled sites

Table 2. Physical and Chemical properties of soil samples collected from different locations of Rawalpindi area.

Sampling sites	EC(dS m ⁻¹)	pH	CaCO ₃ (%)	CEC(C mol _c kg ⁻¹)	Organic Carbon (%)	Clay contents (%)
1) Adiala (Range)	0.35-0.74	7.4-8.0	7.8-21.3	14.7-19.0	0.45-0.85	14-27
Mean	0.47	7.7	14.4	17.2	0.6	21.9
CV±(%)	2.0	0.8	13.5	1.8	8.1	3.4
2) Pirwadhai (Range)	0.29-1.15	7.5-8.1	2.9-20.6	15.4-25.4	0.37-0.97	20-55
Mean	0.70	7.9	10.9	20.4	0.7	33.6
CV±(%)	8.2	0.7	12.2	1.2	6.7	5.6
3) Taxilla (Range)	0.26-0.85	7.6-7.9	7.7-20.8	12.4-19.9	0.51-1.09	10-20
Mean	0.60	7.8	13.5	17.7	0.8	15
CV±(%)	9.2	0.7	14.8	1.3	9.5	4.1
4) Wah factory (Range)	0.25-1.07	7.4-7.8	9.3-17.3	17.5-21.7	0.32-0.64	13-29
Mean	0.62	7.6	13.2	18.7	0.5	20.3
CV±(%)	8.8	0.8	11.7	1.2	8.9	6.9

Table 3. Total heavy metal contents of soil collected from different locations of Rawalpindi (µg g⁻¹)

Sampling sites	Zn	Cd	Fe	Cu	Cr	Pb	Ni
1) Adiala (Range)	30.6-38.4	0.4-1.7	7526-11949	2.2-15.8	1.4-28.8	16.4-28.4	87.6-153.5
Mean	35.3	1.3	9313	10.2	15.9	21.7	128.8
CV±(%)	1.9	19.6	7.1	11.6	12.3	11.3	7.7
2) Pirwadhai (Range)	13.9-200.0	1.7-2.9	13170-24771	12.9-74.2	28.8-124.5	17.8-89.9	25.6-44.0
Mean	69.7	2.2	19645	28.8	70.6	35.8	34.0
CV±(%)	1.6	6.7	0.1	1.4	0.6	0.9	1.4
3) Taxilla (Range)	7.7-30.0	1.9-3.5	16856-26889	20.2-30.2	21.6-144.2	19.4-27.5	19.7-51.6
Mean	20.5	2.7	22549	25.0	81.2	22.8	30.4
CV±(%)	2.5	5.3	0.1	1.2	0.9	0.9	1.3
4) Wah factory (Range)	12.4-197.0	1.9-2.5	22176-30579	10.7-38.2	65.4-154.1	20.3-38.4	28.0-56.9
Mean	67.4	2.1	25183	25.7	114.9	28.2	38.9
CV±(%)	1.8	4.9	0.1	1.4	0.5	1.0	0.9
Permissible limit (a)	80.0	0.5	n/a	20.0	n/a	50.0	25.0
Permissible limit (b)	150-300	1-3	n/a	50-140	100	50-300	30-75
Permissible limit (c)	300-600	3-6	n/a	135-270	n/a	250-500	75-150

Note: n/ a, not available.

a= limits described by Rowell (1994), b = limits described by European community commission (ECC) (1986). c =Permissible limits of Indian standards (Awashti, 2000; Sharma *et al.*, 2006; Gupta *et al.*, 2008).

respectively (Table 3). High mean total Cd values were noticed at Taxilla (2.7 µg g⁻¹) with minimum at Adiala (1.3 µg g⁻¹) sites. Total Fe contents in soils ranged from 7526-30579 µg g⁻¹ (Table 3) with the mean value of 18440 µg g⁻¹. The highest mean total Fe values were observed at Wah factory (25183 µg g⁻¹) and minimum at Adiala (9313 µg g⁻¹) sites. Total Cu concentration varied from 2.2 to 74.2 µg g⁻¹ with mean value of 22.8 µg g⁻¹. Soil samples of Islamabad showed maximum mean total Cu (28.8 µg g⁻¹), whereas the Adiala had minimum (10.2 µg g⁻¹).

Chromium concentrations in soils ranged from 1.4 to 154.1 µg g⁻¹ with mean value of 63.8 µg g⁻¹ (Table 3). Highest mean total Cr values were observed at Wah factory (114.9 µg g⁻¹) and the lowest at Adiala (15.9 µg g⁻¹) sites. In all four locations of Rawalpindi region, total Pb concentrations in soils ranged from 16.4 to 89.9 µg g⁻¹ with mean value of 28.6 µg g⁻¹. Highest value of Pb (35.8 µg g⁻¹) was recorded at Islamabad, whereas minimum (21.7 µg g⁻¹) in Adiala soil samples. Similarly total Ni in soils ranged from 19.7 to 153.5 µg g⁻¹ with average value of 61.4 µg g⁻¹ (Table 3). Highest mean

total Ni values were observed at Adiala ($128.8 \mu\text{g g}^{-1}$), whereas Taxilla sites showed minimum ($30.4 \mu\text{g g}^{-1}$). Overall, the soils of Wah factory and Islamabad contained higher concentrations of total heavy metals as compared to the other two sites.

AB-DTPA-extractable heavy metals: AB-DTPA-extractable zinc concentration ranged from 0.8 to $87.9 \mu\text{g g}^{-1}$ with mean value of $16.6 \mu\text{g g}^{-1}$ (Table 4). Highest mean value was recorded ($27.7 \mu\text{g g}^{-1}$) at Islamabad site, whereas minimum at Adiala ($2.8 \mu\text{g g}^{-1}$). The Cd concentration in sampled sites ranged from 0.0 to $0.49 \mu\text{g g}^{-1}$ (Table 4) with mean values of $0.12 \mu\text{g g}^{-1}$. Highest mean Cd values was observed at Wah factory ($0.20 \mu\text{g g}^{-1}$), whereas minimum ($0.05 \mu\text{g g}^{-1}$) at Adiala sites. AB-DTPA-extractable Fe concentrations in soils ranged from 7.6 to $167.6 \mu\text{g g}^{-1}$ with mean value of ($59.9 \mu\text{g g}^{-1}$). Maximum iron contents were noticed at Wah factory soil samples ($105.7 \mu\text{g g}^{-1}$), whereas minimum at Adiala ($46.7 \mu\text{g g}^{-1}$). Concentration of copper varied from 1.1 to $54.6 \mu\text{g g}^{-1}$ with mean value of $10.8 \mu\text{g g}^{-1}$ (Table 4). Highest Cu was recorded in Islamabad ($17.0 \mu\text{g g}^{-1}$), whereas lowest in Adiala samples ($3.4 \mu\text{g g}^{-1}$). Chromium contents ranged from 0.01 to $1.70 \mu\text{g g}^{-1}$ (Table 4) with maximum value depicted at Wah factory ($0.80 \mu\text{g g}^{-1}$) and minimum at Taxilla ($0.31 \mu\text{g g}^{-1}$). Lead values varied from 2.12 to $38.1 \mu\text{g g}^{-1}$, with maximum value recorded at Islamabad sites ($9.5 \mu\text{g g}^{-1}$) and lowest at Adiala ($4.1 \mu\text{g g}^{-1}$ & $3.3 \mu\text{g g}^{-1}$). Ni concentration varied from 0.2 to $3.8 \mu\text{g g}^{-1}$ with the mean value of $1.2 \mu\text{g g}^{-1}$

(Table 4). Overall results of mean maximum AB-DTPA extractable Ni concentration was recorded in Adiala ($1.9 \mu\text{g g}^{-1}$), whereas lowest ($0.6 \mu\text{g g}^{-1}$) at Taxilla samples. Average Mean values of AB-DTPA extractable Zn, Cd, Cu, Cr, Ni, Fe and Pb at two soil depths showed that all these metals were high in 0-15 cm soil depth than 15-30 cm.

Simple correlation coefficients between AB-DTPA extractable heavy metals and soil physical and chemical characteristics such as pH, EC, CaCO_3 , organic C, clay contents and CEC are shown in Table 5. Soil pH showed negative correlation with AB-DTPA extractable Cr (significant at 5 % and 1%), Pb (significant at 5 %), Fe (significant at 5 %) and AB-DTPA extractable toxic metal (Cd + Cr + Ni + Pb) (significant at 5 %). A positive correlation was observed between EC and AB-DTPA extractable metals Cr (significant at 5 % and 1 %), Pb (significant at 5 % and 1%), Zn (significant at 5 % and 1%), Cu (significant at 5 %) and toxic metals (significant at 5 % and 1%). Results of cation exchange capacity (CEC) revealed negative correlation with only AB-DTPA extractable toxic metals (significant at 5 %). Correlation results of organic C were negative with AB-DTPA extractable Cr (significant at 5 % and 1%), Pb (significant at 5 %) and toxic metals (significant at 5 % and 1%). Percent CaCO_3 depicted negative correlation with AB-DTPA extractable Cr, Pb and toxic metals (significant at 5 % and 1%). Correlation value of percent clay was negative with Cr, Pb and toxic metals (significant at 5 %).

Table 4. AB-DTPA extractable heavy metal contents of soil collected from different locations of Rawalpindi ($\mu\text{g g}^{-1}$)

Sampling sites	Zn	Cd	Fe	Cu	Cr	Pb	Ni
1) Adiala (Range)	0.8-7.6	0.02-0.43	7.6-159.1	1.5-5.7	0.05-0.43	2.4-6.2	0.2-3.8
Mean	2.8	0.10	46.7	3.4	0.30	4.1	1.9
CV±(%)	15.9	19.0	11.0	8.3	11.5	15.3	19.6
2) Pirwadhai (Range)	9.4-87.9	0.00-0.15	11.3-167.6	2.6-54.6	0.01-1.07	2.17-38.1	0.3-1.6
Mean	27.7	0.05	49.9	17.0	0.40	9.5	0.8
CV±(%)	3.4	19.2	1.7	6.1	12.9	2.3	2.8
3) Taxilla (Range)	5.4-18.6	0.03-0.25	12.2-139.4	5.5-18.3	0.11-0.50	2.28-6.97	0.3-0.9
Mean	12.6	0.10	49.1	9.4	0.30	4.1	0.6
CV±(%)	6.2	16.0	1.7	6.1	6.3	2.4	3.6
4) Wah factory (Range)	4.8-30.3	0.04-0.49	21.5-157.1	1.1-15.9	0.14-1.7	2.13-12.1	0.7-1.8
Mean	17.7	0.20	105.7	8.4	0.80	8.3	1.1
CV±(%)	4.9	17.0	2.5	7.4	9.7	1.5	1.9
Permissible limit	*1.5	**0.31	*5.0	*0.50	**8.0	**13.0	**8.1

* = limits described by Soltanpour, (1985); **= limits described by Maclean *et al.* (1987).

Table 5. Simple correlation coefficient of AB-DTPA extractable metals with soil physical and chemical characteristics

Soil Characteristics	Cd	Cr	Ni	Pb	Fe	Zn	Cu	Toxic metals
pH	-0.22	-0.51**	-0.15	-0.41*	-0.48*	-0.27	-0.22	-0.45*
EC	0.03	0.62**	0.04	0.69**	0.33	0.56**	0.47*	0.72**
CEC	0.11	-0.32	-0.35	-0.36	-0.17	-0.17	-0.24	-0.41*
CaCO ₃ %	0.25	-0.51**	-0.11	-0.53**	-0.35	-0.40*	-0.43*	-0.55**
Organic C	-0.11	-0.63**	-0.21	-0.46*	-0.20	-0.33	-0.09	-0.51**
Clay (%)	-0.04	-0.42*	-0.15	-0.40*	-0.31	-0.28	-0.39	-0.43*

(*) significant at 5% and (**) at 1% probability levels, respectively.

DISCUSSION

The EC of effluent samples used for irrigation at 23 sampled sites was above the critical limits of 0.70 dSm⁻¹ as described by Ayers and Westcot (1985). Waste water samples of 10 sites were above the critical limit of 1.5 dSm⁻¹ as described by WWF (2007) (Table 1) for Pakistan. Overall samples showed high EC and the causes could be the salts added by textile mills, laundries, residential and factory wastes (Murtaza *et al.*, 2008). All the soils irrigated with wastewater in the study showed EC in safe range, whereas in contrast to this some other scientists reported high EC and pH of soils irrigated with untreated wastewater (Lone *et al.*, 2000; Murtaza *et al.*, 2008). The reasons are high EC and pH of the effluents being used for irrigation of those soils as compared to present sites. Moreover, our sampling sites fall in the region of semiarid to sub humid, which resulted in low EC of soils as compared to wastewater irrigated soils of low rainfall areas. In 24 effluent samples, TDS value was above the toxic limit (450 mg l⁻¹) as described by Ayers and Westcot (1985), whereas 10 sites were above the limits of WWF (2007) (Table 1). Maximum mean TDS, SAR and EC values was noticed in effluent samples of Islamabad/ Pirwadai site, which were higher than toxic limits. The combination of high EC and SAR of the raw effluents could be slight to moderately hazardous for most soil textures according to the criteria for irrigation water quality (Ayers and Westcot 1985). Thus, the long-term use of these effluents for irrigation purpose should be discouraged, because their continuous use will be harmful for soil health (Awan *et al.*, 2002; Sial *et al.*, 2006). The pH of 24 effluent samples was within the permissible limits (6.5-8.4) of Ayers and Westcot (1985) and WWF (2007), whereas only one sample of Adiala site showed high pH (8.6). On the whole, pH of soils did not appear to be problematic even after long time raw effluent irrigation, most probably because of the regular addition of organic matter and soil calcareousness. Effluents pH values were within

permissible limits but continuous application of alkaline or high pH effluents will make the soils unfit for further cultivation, as the pH of most of our soils is already quite high (Sial *et al.*, 2006; Murtaza *et al.*, 2008). However, the buffering capacity of soil tends to bring homeostasis and lowers the pH of applied effluents in line with the pH of the soil. The values of residual sodium carbonate (RSC) in all the effluent samples were below the safe limit of Ayers and Westcot (1985) (2 meq l⁻¹); however, based on the criteria of WWF (2007) (1.25 meq l⁻¹) 3 effluent samples had higher RSC than the safe limit. Thus, overall the effluents under study could be used safely for irrigation purpose as far as their RSC is concerned.

Heavy metal contents in wastewater samples used for irrigation in farmers field were compared with critical levels described by Ayers and Westcot (1985) and WWF (2007) water quality permissible limits for Pakistan (Table 1). Limits described by FAO and WWF are similar for Zn, Fe, Ni, Cd & Cu whereas values defined for Cr and Pb by FAO are higher than those of WWF. The amount of Zn present in the effluents was in the order Wah factory > Adiala > Islamabad/ Pirwadai area > Taxilla sites. In all effluent samples, Zn and Pb were within permissible limits (2.0 µg ml⁻¹) (Table 1) of Ayers and Westcot (1985), and WWF (2007). The, Cd contents in 21 effluent samples were above the permissible limit (0.01 µg ml⁻¹) but showed the same order of occurrence like Zn i.e., Wah factory depicting higher concentration than Adiala, Taxilla and Islamabad sites. Iron contents in the 22 effluents samples were within permissible limit of 5 µg ml⁻¹ (Ayers and Westcot, 1985; WWF, 2007) except 2 samples from Adiala sites and one from Wah factory area showed values above permissible limits. Copper concentration in effluents sample of five sites was above the critical limit of 0.2 µg ml⁻¹ (Ayers and Westcot, 1985; WWF, 2007). Concentration of Cr in 19 effluent samples was higher than 0.1 µg ml⁻¹ (Ayers and Westcot, 1985), whereas all the effluent samples were higher than limit (0.01 µg ml⁻¹) described by WWF (2007). Nickel in fourteen effluent samples was above

the permissible limit of $0.2 \mu\text{g ml}^{-1}$ (Ayers and Westcot 1985; WWF 2007). Ten effluent samples showed concentration of Pb higher than $0.1 \mu\text{g ml}^{-1}$ (WWF, 2007), whereas all the samples were in safe limit ($5 \mu\text{g ml}^{-1}$) described by Ayers and Westcot (1985). Our findings are in line with those of Latif *et al.* (2008) and Gulfraz *et al.* (2002) who too observed heavy metals concentration in sewage water used by farmers for irrigation in Rawalpindi area above critical limits. Research carried out in Faisalabad (Murtaza *et al.*, 2008) and in Haroonabad (Ensink *et al.*, 2004) also indicated higher concentrations of heavy metals in effluents used for irrigation in those areas. It was further revealed that long term use of such effluents for irrigation may cause accumulation of toxic metals in soils, which may lead to their elevated levels in plants through bioaccumulation. Consumption of such plants may pose a potential threat to animal health (Ensink *et al.*, 2004). Moreover, accumulation of heavy metals in soil reduces the size of soil microbial biomass and results in development of metal tolerant bacterial species (Clemente *et al.*, 2007). In addition to this entry of untreated wastewater into the rivers and lakes is also threat for aquatic life (Javed and Hayat, 1998, 1999).

Total heavy metal contents in farmers field were compared with critical levels described by Rowell (1994), European community commission (ECC) (1986) and Indian standards (Awashthi, 2000) (Table 3). AB-DTPA extractable heavy metal contents in farmers field were compared with critical levels described by Maclean *et al.* (1987) for Pb ($13 \mu\text{g g}^{-1}$), Cd ($0.31 \mu\text{g g}^{-1}$), Cr ($8.0 \mu\text{g g}^{-1}$), Ni ($8.1 \mu\text{g g}^{-1}$), and with Soltanpour (1985) for Cu ($0.50 \mu\text{g g}^{-1}$), Fe ($13 \mu\text{g g}^{-1}$) and Zn ($1.5 \mu\text{g g}^{-1}$) (Table 4) contents in soils. Total Zn concentration, when compared with permissible limit of Rowell (1994) was much higher in soils of Islamabad and Wah factory, whereas the soils of Taxilla and Adiala were in safe range. However, the Total Zn concentration was within permissible limits in all the soils when compared with ECC ($150\text{--}300 \mu\text{g g}^{-1}$) and Indian standards ($300\text{--}600 \mu\text{g g}^{-1}$). Overall AB-DTPA extractable Zn concentration was in the order: Islamabad > Wah factory > Taxilla > Adiala being much higher than the permissible limit in 24 soils under study. Both Total and AB-DTPA heavy metals value were high in Islamabad soil samples. In addition to this, AB-DTPA extractable Zn as a fraction of total Zn in soils showed highest Zn value (61.5%) in Taxilla soils while minimum in Adiala soils (7.9%).

Total Cd concentration in 24 soils was found higher than critical limit of $0.50 \mu\text{g g}^{-1}$ (Rowell 1994), except one soil at Adiala where concentration of cadmium was lower ($0.4 \mu\text{g g}^{-1}$) than permissible limits. Comparison

with ECC and Indian standards showed concentration in 4 soils above the ECC limits ($1\text{--}3 \mu\text{g g}^{-1}$) whereas all were within range according to Indian standards ($3\text{--}6 \mu\text{g g}^{-1}$). In twenty three soils, AB-DTPA extractable Cd concentration was below the critical limit of $0.31 \mu\text{g g}^{-1}$ (Maclean *et al.*, 1987) except two soils, one at Adiala and the other at Wah factory showed concentration of cadmium higher than permissible limits. Murtaza *et al.* (2008) studied Co, Cd and Mn in soils at Faisalabad irrigated with city effluents, who also found AB-DTPA extractable Cd below the permissible limits. They further calculated the time required by soil to reach the loading limit, which revealed Cd concentration between 0.88 and 0.96 kg ha^{-1} that was double the annual input limit of 0.5 kg ha^{-1} as proposed by US EPA (Page and Chang, 1985). Since retention of Cd in soil is high and is not leached easily because of which total Cd will increase in future if similar practice is continued. The low concentration of AB-DTPA extractable Cd could be due to formation of Cd complex with clay minerals and humic substances in the alkaline soils. Highest extractable Cd fractions was recorded at Wah factory (9.5%), whereas minimum at Islamabad (2.3%). AB-DTPA extractable and total iron contents were highest at Wah factory as compared to all other sites. All the soils had Fe content higher than the permissible limits. Available Fe fraction in comparison to total Fe contents were in the order: Adiala (0.50 %) > Wah factory (0.42 %) > Islamabad (0.25 %) > Taxilla (0.22 %). AB-DTPA extractable copper concentration was above the critical limit ($0.5 \mu\text{g g}^{-1}$) in all the sampled sites, while fourteen soils samples showed total copper higher than permissible limit ($20 \mu\text{g g}^{-1}$). Data of total Cu was in permissible limit when compared with ECC ($50\text{--}140 \mu\text{g g}^{-1}$) and Indian standard ($135\text{--}270 \mu\text{g g}^{-1}$). From all sites, high quantity of AB-DTPA extractable and total copper was observed at Islamabad/ Pirwadai site. Available Cu fractions were in the order: Islamabad (59 %) > Taxilla (38 %) > Adiala (33 %) > Wah factory (33 %). Seven soil samples of Wah factory, Taxilla and Islamabad showed total Cr above the permissible limits of ECC ($100 \mu\text{g g}^{-1}$) whereas all soil samples showed AB-DTPA extractable Cr within permissible limit ($8.0 \mu\text{g g}^{-1}$). Overall AB-DTPA extractable and total Cr in sampling sites was in the order of Wah factory > Taxilla > Islamabad > Adiala. Available Cr fractions were in order: Adiala (1.9%) > Wah factory (0.7%) > Islamabad (0.6%) > Taxilla (0.4 %). Twenty-three soil samples showed total Ni contents higher than permissible limit of $25 \mu\text{g g}^{-1}$ (Rowell, 1994), whereas 7 soils of Adiala were higher than ECC limits ($30\text{--}75 \mu\text{g g}^{-1}$). Only one soil showed total Ni higher than Indian standards ($75\text{--}150 \mu\text{g g}^{-1}$). AB-DTPA extractable Ni contents of all soil samples were within permissible

limit of $8.1 \mu\text{g g}^{-1}$. Overall, mean total and extractable Ni values of Adiala soils were higher than the other sites. Highest available fractions were observed at Wah factory (2.8%), whereas minimum at Adiala (1.5%). Regarding Pb only one site at Islamabad showed total & AB-DTPA extractable Pb above the permissible limit of $50 \mu\text{g g}^{-1}$ (Rowell 1994) and $13 \mu\text{g g}^{-1}$ (Maclean *et al.*, 1987). All the sites had lead within the safe limit of ECC ($50\text{-}300 \mu\text{g g}^{-1}$) and Indians standards ($250\text{-}500 \mu\text{g g}^{-1}$). The highest available Pb fraction was observed in soils of Wah factory (29.4%), while the minimum at Taxilla (17.9%).

Overall, the results showed high concentrations of heavy metals in effluents of Adiala area, whereas the metal concentrations (AB-DTPA extractable & total heavy metals) were higher in soils of Wah factory and Islamabad. It is probably because of the more build up of heavy metals in the soils of these two sites. Accumulation of heavy metals in soils depends on a number of factors such as the organic matter addition through root activities and crop residues, calcareousness of the soil and high proportion of clay minerals carrying pH-dependent charges (Murtaza *et al.*, 2008). The concentrations of heavy metals in effluent samples of Wah factory and Islamabad were higher next to those of the Adiala area. Thus, a combination of soil and effluent composition factors might be the cause of higher accumulation of heavy metals in soils of Islamabad and Wah factory area. These results are contradictory to those of Latif *et al.* (2008) who found AB-DTPA extractable metals in permissible limits, whereas at certain locations in our study heavy metals were higher than permissible limits. A similar study conducted at Lahore, Pakistan (Kashif *et al.*, 2009) also revealed high DTPA extractable heavy metals and high metal contents in vegetables grown with wastewater. Overall, in our soils AB-DTPA extractable fractions of heavy metals in comparison to total metal contents in soils were quite low which appears to be due to high pH and Calcium carbonate contents in our soils, resulting continuous immobilization of metals in soils (Lee *et al.*, 2004). Ensink *et al.* (2004) surveyed farmers using wastewater for irrigation in Faisalabad and Haroonabad, Pakistan and found total Cr, Ni, Cd, Pb, Cu & Zn in soils within permissible limits, whereas high levels of heavy metals were found in plant samples. They suggested regular monitoring of total metal contents of soil, which otherwise would reach levels toxic to plants and soil biological functions. It is further added here that data ranges of measured total heavy metals in all our sites were higher than those observed at Faisalabad and Haroonabad.

It is well known that metals solubility in soils mainly depends on soil pH, organic C, CEC, and clay contents (Hough *et al.*, 2003; Walker *et al.*, 2003). Therefore, in the present study, effects of soil properties on bioavailable fraction (AB-DTPA extractable) of heavy metals were studied (Table 5). Results showed that soil pH, organic C, CEC, clay contents, and CaCO_3 had negative effects on the extractable fractions of heavy metals in soils, although increase in soil AB-DTPA extractable metals enhanced soil EC. These results are in line with Rattan *et al.* (2005), who showed negative correlation of soil pH with DTPA extractable metals, but are contradictory to his findings of soil organic C, which were positive with respect to DTPA metals in soils. Perez-de-Mora *et al.* (2005) observed decrease in soluble heavy metal concentrations with increase in organic matter contents of soil. Organic materials contain high concentrations of stabilized humic substances, which can influence metal availability by adsorption and forming stable complexes. Clemente *et al.* (2005) reported that organic materials in soil not only improve soil fertility, but also change heavy metals availability and thus can be used in reclamation process. Similarly, Derome and Saarsalami (1999) and Puschenreiter *et al.* (2005) reported decrease in heavy metal availability with the increase in CaCO_3 contents in soil and recommended lime application to metal contaminated soils to decrease metal availability to plants and soil microbes and to reduce their transfer into the food chain. Sipos *et al.* (2005) reported 50% reduction in Pb retention capacity of soils with decrease in organic matter and calcium carbonates contents of soil. Soils high in clay-sized particles have a tendency to adsorb higher amounts of trace metals than the coarse textured soils due to their high specific surface area and more CEC, which create unique conditions in the fine textured soils for trapping of incoming metal pollutants. Francois *et al.* (2004) reported that soils with high clay, organic matter, CaCO_3 contents and moderately alkaline pH had low metal mobility and bioavailability due to high binding capacity. Coarse textured soils with low organic matter contents generally possess poor metals retention capacity and ultimately give rise to higher metal concentrations in soil solution (Moller *et al.*, 2005).

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