HEAVY METALS CONCENTRATION IN SELECTED SOIL SAMPLES COLLECTED FROM HARIPUR, KHYBER PAKHTUNKHWA

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ABSTRACT: *Wastewater irrigation is becoming a common practice in low income agricultural countries like Pakistan. With repeated application of wastewater, the composition of agricultural soil is continuously modified. The current study is aimed to assess the effects of applying wastewater to cultivated land of Haripur region, Pakistan. The concentration of selected heavy metals such as copper (Cu), zinc (Zn), cadmium (Cd), chromium (Cr), nickel (Ni) and lead (Pb) were find out at two different cultivated fields using Atomic Adsorption Spectrophotometer. The results revealed that the concentration of Cu was 1.26 $mgkg^{-1}$, Zn 0.43 $mgkg^{-1}$, Cr 1.62 $mg\ kg^{-1}$, Cd 1.18 $mg\ kg^{-1}$, Ni 2.95 $mg\ kg^{-1}$, and Pb 4.9 $mg\ kg^{-1}$ in clay loam and silt loam soil with pH range of 7.2-7.8. Lead was observed to be more in concentration as compared to Ni followed by Cr, followed by Cu, followed by Cd and Zn. The concentration of all the selected heavy metals in wastewater irrigated fields were higher in comparison to control fields at ($p<0.01$). The increase in the concentration of heavy metals due to wastewater irrigation may have negative impacts on crops productivity as well as on the health of the producers and consumers. Campaigns vis-à-vis awareness about the use of wastewater as a resource in agriculture with comprehensive monitoring system of soil quality are some of the policy recommendations extended in the light of this study.*

Keywords: Wastewater Irrigation; Agricultural soil; Heavy metals.

1. INTRODUCTION

Wastewater can be defined as low quality water that might threaten agriculture sustainability as well as human health, but it may be safe for using in irrigation agriculture provided that certain precautions are taken. It portrays water which has been polluted due to mixing with effluents or agriculture run-off [1]. Wastewater is becoming a major source of water in urban agriculture system in order to raise the socioeconomic conditions of the local community. In developing countries like Pakistan, most of the urban centers have no waste water treatment facilities and 2% of the total population has access to the sewage networks [2, 3]. So the generated wastewater is fed into surface water bodies such as irrigation channels. However this water is being used to irrigate some 37000 hectares of land, contributing about 26% of the total national crop production [4]. There exist no regulations for the utilization of wastewater for irrigation purposes within the country. Farmers generally prefer wastewater because of high nutrient value and free of cost and continuous supply round the year.

Wastewater in urban areas is generally run off in surface water bodies like rivers and streams that may also contain industrial waste as well as heavy metals. Heavy metals present in wastewater generally get absorbed in soil and can be retained for longer period of time because of non biodegradability and non thermo degradability. Each soil is unique in its characteristics and changes in its perfunctory properties like pH, clay and organic matter content, cation exchange capacity and ions holding capacity would lead to release of heavy metals available for plant uptake [5]. The organic matter supplied by wastewater irrigation to the soil is eventually decomposed to low molecular weight soluble organic acids, residual organic matter and inorganic constituents that may alter the soil composition and structure which result in the accumulation of metal on soil surface. Repeated application of wastewater to cultivated land my lead to the accumulation of heavy metals upto the levels that are toxic for plant growth as well as for health of consumers.

This work assumed that wastewater may be a combination of agricultural runoff, domestic sewage, human and animals waste, black and gray water, and wastewater from residential and commercial establishments. The objectives of the study were check the soil irrigated with wastewater for heavy metals concentration. The heavy metals such as copper (Cu), zinc (Zn), cadmium (Cd), nickel (Ni), lead (Pb) and chromium (Cr) were considered in our study. This paper is aimed at supplementing the existing databy providing more recent measures of heavy metals contamination due to wastewater irrigation [6, 7]. This would add to existing knowledge on wastewater irrigation and its impacts soil quality.

2. MATERIALS AND METHODS

2.1 Site description

In current study three vegetables production sites i.e. Noor colony, Naseem town and Poli stop, were selected from Haripur, Khyber Pakhtunkhwa, Pakistan (Figure 1). These sites were selected on the basis of pollution activities and or high agro-economic activities practiced in the region. The concentrations of heavy metals were determined in soil at two vegetable production sites where wastewater has been used for irrigation for at least 30 years.The canal flows through industrial and residential areas of Haripur receiving waste discharges at various points as we move downstream. Increased industrial and residential structures result in the release of unknown toxic chemicals and effluent into the canal. Site Poli stop was irrigated with ground water. In general, bed and furrow irrigation system is being practiced in the waste water irrigated fields with average depth of 65cm with biannual cultivation.The crops grown at three studied sites include leafy vegetables like, cauliflower (*Brassica oleracea),* Radish (*Raphanus sativus*), tomatoes (*lycopersicon esculentum*), lady finger (*Abelmoschus)*, Pea (*Cicer arietinum*), Garlic (*Allium sativum)* and Onions (*Alluim Cepa*). These vegetables are produced for domestic use as well as for trade in local markets.

2.2 Soil Sampling

For the analysis of different heavy metals, soil samples were collected randomly using Dutch auger method at different depths of 00-12cm, 12-24cm and 24-36cm both in waste water irrigated and ground water irrigated lands. A total of 10 soil samples were collected randomly with nine samples from waste water irrigated field and one sample from ground water irrigated area. Soil samples taken from different depths also indicated the layering pattern of soil in the studied area. Basic Properties of the studied soils are presented in (Table 1).

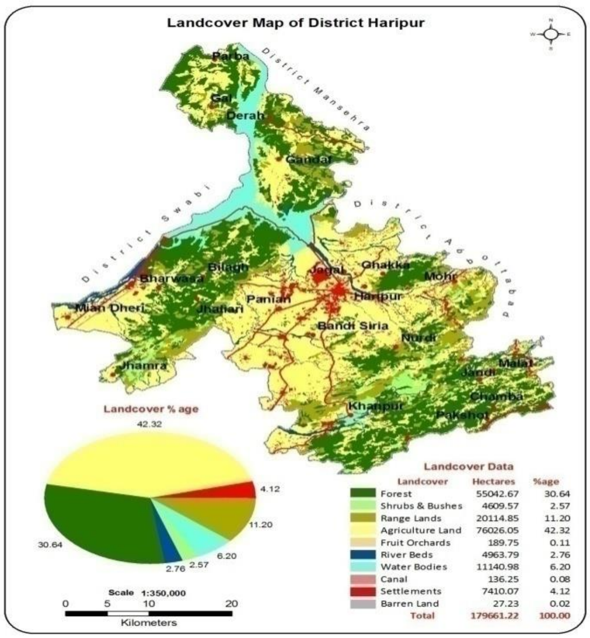

Figure 1: Map of physical features of Haripur region, Pakistan.

2.3 Soil analysis

Different tests were carried out in order to determine the physiochemical properties of soil. Water method was used for determination of soil reaction while Electrical conductivity was measured using electrical conductivity meter[8]. Hydrometer method was used to determine Soil texture[9]. Lime was determined by acid neutralization method was followed for determination of lime content of the soil [10].Chan *et al.*,[11] suggested method using standardized solution of $FeSo_4$ and $Cr_2K_2O_7$ was adopted for Soil organic matter determination. Ammonium bicarbonate diethylene tri Amin penta acetate (AB-DTPA) or Mehlic No.3 extractable P and K was determined on the basis of pH of the soil samples collected from the study area. In case of low pH (7 and below) Mehlic -3 extractant was used. AB-DTPA extractant was used in case of pH (7 and above).

2.4 Heavy metals analysis

Soil samples from different sites were analyzed for heavy metals using Baker, *et al.*, 1982 suggested method, the aqua regia digest method [12]. One gram of soil for each sample, was transferred into a 100 mL digestion flask and 10 mL of aqua regia (a mixture of concentrated HCl and concentrated HNO_3 in the HCL: HNO_3 ratio of 3:1) was added and the mixture was allowed to react overnight (for at least 12 hours). The next day, mixture was heated with time and boiled under reflux for 2 hours after which the digestion flask was cooled. The cooling column was rinsed with 15 mL of distilled water recovering rinse water in the digestion flask. The mixture was separated using a centrifuge at 1500 rpm for 5 min after which a supernatant solution was collected into 50 mL volumetric flask before diluting to the mark with hot 2 M HNO_3. The soil extract was analyzed for Cu, Zn, Cr, Cd, Ni and Pb usingan atomic adsorption spectrophotometer (model: Philips AA-10). Standard solutions were prepared in the concentration ranges of 0-5 mg/L for Cu, Zn, Ni, Cr and Pb, and 0-1 mg/L for Cd.

2.5 Statistical analysis

Data were subjected to statistical package (SPSS-17) to compare any significant difference present between wastewater irrigated soil and control soil.

3. RESULTS AND DISCUSSIONS

3.1 pH

Most of the crop nutrients and potential toxic constituents highly depend on the level of pH in irrigation water. At higher pH, the nutrients and heavy metals are more difficult to be extracted than in lower pH condition. Irrigation water affects the soil pH slowly, so the effect of soil pH on crop production is indirect. Too low and too high pH of irrigation water in contact with crop leaves will damage the crop and influence the yield. The mean pH of the soil at wastewater irrigated sites (Noor colony and Naseem town) and control site (Poli-stop) with their standard deviation (SD) is given in the (Table 2). Soil samples collected from wastewater irrigated fields showed the lowest pH that is 6.93, while the highest pH (7.27) was recorded for soil samples collected from wastewater irrigated fields. Wastewater irrigated soil showed higher pH in comparison to control soil.

Soil pHwas increased (from 6.93 to 7.27) by 0.34 units at Noor colony and Naseem town site due to wastewater irrigation when compared with control soil. The results also showed that the pH of the first 00-12 cm layer of wastewater irrigated soil was high as compared to control soil. It can be inferred from the results that the wastewater application has slightly raised the soil pH.

3.2 Clay Content

The mean clay content of the wastewater irrigated sites and ground water irrigated site with their standard deviation (SD) is given in (Table 2). The highest Mean clay content (28 %) was shown by the soil samples collected from wastewater irrigated fields. The least clay content (23.46 %) was revealed by the soil samples collected from Naseem town. The clay content in the third 24-36 cm layer of wastewater irrigated soil samples collected from Naseem town was low (21.2%) as compared to soil samples collected from Poli-stop as well as Noor colony that is (30.8%). It can be inferred from these results that as the depth of the soil increases the clay content also increases but it is not true for the soil samples collected at 00-12cm layer from Poli-stop as this site showed the least (19.2 %) clay content.

3.3 Copper (Cu)

Copper is an element for animals and plants growth. Sewage sludge, pesticides, and fertilizers are the most common sources of copper in soils. Irritation of the upper respiratory tract, metallic taste in mouth and nausea are among the diseases that are caused by Copper dust [13]. The mean concentration($mgkg^{-1}$) of Cu along with its SD is given in (Table 2). The concentration 1.26 mg/kg of copper was found high in the soil from wastewater irrigated sites. The soil samples collected from ground water irrigated site showed lowest concentration of Cu which is 1.23 mg/kg. The concentration of copper in soil samples collected from Naseem town was higher followed by Noor colony, followed by Poli-stop. The application of wastewater had augmented the soils with copper because the results revealed that the concentrations of the selected heavy metal in soil irrigated with wastewater were above the concentration recoded for control soil.

3.4 Cadmium (Cd)

Cadmium (Cd) is a major health concern because of its toxicity. Phosphate fertilizer, non-ferrous smelters, lead and zinc mines and irrigation with wastewater are most widespread sources for cadmium in soil and different parts of plants [14]. Micronutrients and heavy metals accumulation could be caused directly by the wastewater composition or indirectly through Chelation and acidification effects of the applied wastewater [15].Pb concentration in ($mgkg^{-1}$) along with its SD at control site and wastewater irrigated site is given in (Table 2). The highest mean concentration (0.38$mgkg^{-1}$) of Cd was found in soil samples collected from Noor colony followed by Naseem town 1.18 ($mgkg^{-1}$) and Poli-stop 0.13 ($mgkg^{-1}$). The first 00 to 12 cm layer of soil at wastewater irrigated site Naseem town showed higher concentration than the average concentration of Cd in control soil. Wastewater irrigation caused an increase of Cd in soil. The results indicate that the surface layers of wastewater irrigated soils contain greater amount of Cd in comparison with subsurface layers and control soils.

3.5 Chromium (Cr)

Chromium is among certain notorious environmental pollutants in the world, known to have toxic effects on soil flora and fauna. Chromium contamination occurs due to sewage sludge application and fly ash, tanneries and steel industries. The most likely sources of chromium are Motor vehicles [16]. Critical limits of chromium for plants ranged from 5 to 30 ($mgkg^{-1}$). Higher concentration of chromium may also result in reduction of crops yield. Skin blistering, stomach ache, kidney and liver damage, alteration of genetic material, lung cancer and ultimately death are the likely problems associated with exposure to chromium [17]. The mean concentrations ($mgkg^{-1}$) of Cr at wastewater irrigated soil and ground water irrigated soil with their SD is given in (Table 2). Significantly different amounts of Cr were shown by Soil samples collected from three different vegetables and crop production sites. The highest average concentration (1.62$mgkg^{-1}$) was recorded for wastewater irrigated soil collected from Naseem town. Ground water irrigated soil showed the least average concentration (1.22$mgkg^{-1}$) of chromium.

3.6 Zinc (Zn)

Zn is essential plant micronutrients. However, at high levels, they may be toxic to plants. Inhalation of Zn may cause diseases viz-a-viz throat dryness, sweat taste, cough, weakness, aching, fever, nausea and vomiting. Zink addition to the diet accelerates the growth of delayed sexual development and plays a vital role in wound healing, while its shortage causes loss of sense of touch and smell. The mean concentrations ($mgkg^{-1}$) of Zn at three studied sites with their SD is given in (table 2). The highest average concentration (0.43$mgkg^{-1}$) of zinc was found in soil samples from Noor colony while the lowest (0.33$mgkg^{-1}$) was found in soil samples from Poli-stop. The least (0.33$mgkg^{-1}$) concentration of Zn was found in soil samples collected from groundwater irrigated site. The magnitude of contamination was in the order Noor colony > Naseem town > Poli-stop. The wastewater applied to soil enhanced the concentration of zinc (Table 2).The results showed that the soil irrigated with wastewater had the highest concentration of Zn as compared to ground water irrigated soil.

3.7 Lead (Pb)

Plants and animals and especially microorganisms are prone to lead toxicity. Lead is generated mainly by sources like fuel combustion, sewage sludge application and farmhouses. Vehicular emission of Lead from gasoline can also be deposited on soil. Long term exposure to Pb can develop symptoms like anemia, pale skin, a decrease handgrip strength, abdominal pain, nausea, vomiting and paralysis of wrest joint. Continued exposure can lead to decrease fertility and/or increase chances of miscarriages or birth defects.

The mean concentrations ($mgkg^{-1}$) of Pb at wastewater irrigated sites and ground water irrigated site with their SD is given in (Table 2). Average lead concentrations were found high in wastewater irrigated soil collected from Naseem town. The highest concentration (5.64 $mgkg^{-1}$) of Pb was recorded in the first 00 to 12 cm layer of control soil at Naseem town. Soil samples from Noor colony showed the least average concentration (1.03$mgkg^{-1}$) of Pb. The concentration of Pb in soil samples collected from Poli-stop showed the greatest SD from wastewater irrigated sites (Noor colony and Naseem town).

The results showed that wastewater irrigated soil had higher concentration of Pb. overall the concentrations were generally low at this site. Higher concentrations of Lead (3.2 mgKg^{-1}) in control soil relative to wastewater-irrigated fields (1.03 mg Kg^{-1}) at the Noor colony site.

3.8 Nickel (Ni)

Nickel is an abundant element. Volcanic eruptions give rise to Ni found in all soils. Nickel itch is the most common ailment arising from nickel or its compound. Nickel is carcinogenic in nature. It has adverse effects on nasal cavities and lungs.

Table 2 shows mean concentrations ($mgkg^{-1}$) of Ni at wastewater irrigated soil and ground water irrigated soil. The concentration of nickel was high in soil samples collected from Naseem town and Noor colony and least in Poli-stop area. The average concentration of Ni was found high in wastewater irrigated soil than control soil (Table 2). All time's high concentration of Ni such as (2.47±0.21$mgkg^{-1}$) was shown by soil samples collected from Naseem town, which is located downstream of the irrigation canal. The lowest concentration of Ni such as 1.03±0.006 $mgkg^{-1}$ was

Table 1: Basic physico-chemical properties of soils from wastewater-irrigated fields and ground water irrigated fields (control soil) at District Haripur, Khyber Pakhtunkhwa, Pakistan

Characteristics	Noor colony (A) Wastewater irrigated site			Naseem town (B) wastewater irrigated site			Poli stop(C) Control/freshwater-irrigated site		
Soil depth(cm)	00-12	12-24	24-36	00-12	12-24	24-36	00-12	12-24	24-36
Clay (%)	23.2	30	30.8	32	17.2	21.2	19.2	27.2	30.8
Silt (%)	55.2	49.2	53.2	47.2	67.2	51.2	59.2	51.2	49.2
Sand (%)	21.6	20.8	16	20.8	15.6	27.6	21.6	21.6	20
Texture	Silt loam	Clay loam	Silt loam	Clay loam	Silt loam	Silt loam	Silt loam	Clay loam	Clay loam
EC ms/cm	0.299	0.094	0.18	0.117	0.167	0.282	0.095	0.207	0.05
OM (%)	1.035	1.035	0.69	1.38	0.69	1.035	1.035	0.82	0.69
PH	7.6	7	7.2	7.4	7.2	7.2	6.8	7	7
Lime (%)	6	5	5	5	5	6	6	5	2.5
N (%)	0.0517	0.0517	0.0345	0.069	0.0345	0.0517	0.0517	0.0144	0.0345
P (mg/kg)	5	5	5	5	4	5	5	5	4
K (mg/kg)	76	132	80	154	84	130	70	90	86

Table 2: Heavy metals concentrations with their standard deviation and mean values

HM (mg/kg)	Site											
	Noor colony				Naseem town				Poli-stop			
Soil depth	00-12cm	12-24cm	24-36cm	Mean±SE	00-12cm	12-24cm	24-36cm	Mean±SE	00-12cm	12-24cm	24-36cm	Mean±SE
pH	7.6	7	7.2	7.27±0.3	7.4	7.2	7.2	7.27±0.11	6.8	7	7	6.93±0.11
Clay (%)	23.2	30	30.8	28±4.17	32	17.2	21.2	23.46±7.65	19.2	27.2	30.8	25.73±5.93
Cu	1.206	1.284	1.124	1.24±0.04	1.418	1.21	1.16	1.26±0.14	1.228	1.436	1.032	1.23±0.2
Cd	0.124	0.9	0.124	0.38±0.45	1.52	1.12	0.912	1.18±0.31	0.138	0.13	0.058	0.13±0.01
Cr	0.98	0.7	2.4	1.36±0.91	1.26	1.87	1.62	1.62±0.32	1.5	1.26	1.22	1.22±0.3
Zn	0.63	0.338	0.32	0.43±0.17	0.388	0.346	0.46	0.4±0.06	0.366	0.231	0.39	0.33±0.09
Pb	0.74	1.24	1.12	1.03±0.26	5.64	4.24	4.82	4.9±0.7	3.6	2.8	1.96	3.2±0.57
Ni	1.02	0.82	1.14	0.99±0.26	2.26	3.7	2.88	2.95±0.72	1.58	1.74	1.98	1.66±0.11

Table 3:Variation in concentration of heavy metals in control soil and wastewater irrigated soil (upstream and downstream)

	Test Value = 0					
					95% Confidence Interval of the Difference	
	t	df	Sig. (2-tailed)	Mean Difference	Lower	Upper
Upstream	1.510	7	.175	5.08750	-2.8785	13.0535
Control soil	1.988	7	.087	5.38000	-1.0194	11.7794
Downstream	1.656	7	.142	5.05375	-2.1647	12.2722

found in soil samples collected from the control site. The concentration of nickel in soil was in the order Naseem town> Noor colony > Poli-stop. The results also showed that the Nickel concentration in soil irrigated with wastewater was high in comparison to control soil (Table 2).

4.CONCLUSIONSAND RECOMMENDATIONS

Due to non-availability of fresh water for irrigation, farmers in Haripur are highly dependent on wastewater as a main production factor and life line for their livelihood. The study concludes that pH of wastewater irrigated soil and control soil was within the neutral range. The soil showed silt loam and clay loam texture. Under the current conditions of generally neutral to alkaline pH, silt loam and clay loam textured soils. It was concluded that the concentrations of Cu, Zn, Cd, Cr, Pb and Ni were high in enrichment of the heavy metals were in the sequence: Pb (4.9 mgKg^{-1}) > Ni (2.95 mgKg^{-1}) > Cr (1.62 mgKg^{-1}) > Cu (1.26 mg Kg^{-1}) > Cd (1.18 mg Kg^{-1}) > Zn (0.4 mg Kg^{-1}). It was concluded that soil contamination due repeated application of wastewater to irrigate fields may pose a threat to environmental sustainability and human survival. The study recommended development of urban farming on a sustainable basis through proper planning and management under proper policy framework for urban development. Such policies and interventions should be enlightened from the experiences of the farmers and rich literature available from all over the world. Some of the basic components of such interventions should include development of comprehensive monitoring system, awareness raising and public participation at large.

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