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Assessment of industrial effluents for heavy metals concentration and evaluation of grass (*Phalaris minor*) as a pollution indicator

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A R T I C L E I N F O

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A B S T R A C T

This study was conducted to investigate the impact of industrial activities on heavy metals status in wastewater, sludge and flora on the bank of selected main drains of the Hayatabad Industrial estate, Peshawar. Plants, sludge and wastewater samples of selected sites were collected and analyzed for heavy metals distribution; cadmium (Cd), chromium (Cr), lead (Pb) and zinc (Zn) levels. Bioconcentration factor (BCF) values were calculated for plants (*Phalaris minor*) grass species found naturally at all sites. The results showed that the levels of metals in wastewater were lower than permissible limits except Cd and the concentration of metals in plants and sludge were within permissible limits when compared to their respective standards. Metal distribution was in the following order; sludge > plants > wastewater and the concentration of metals varied according to the distance from the source with no specific pattern. Sludge samples for all sites showed a high concentration of metals as compared to plants and wastewater samples. In grass samples, Zn was highest and Cd was low for all sites. Metals accumulation in plants was in order of; roots > shoot. Pearson's coefficient correlation showed that Cr in plant roots and Zn in shoots showed significantly high correlation with Cd in sludge while Pb in roots showed significant negative correlation with Zn in sludge. BCF values for Cr, Pb and Zn were >1, showing the phytoremediation potential of plants.

1. Introduction

Heavy metals occur naturally in soil, water, air and various organisms in low concentrations. If its level exceeds certain limits, it may cause a number of ecological and health problems [1,2]. Heavy metals are famous for toxicity, persistence in the environment (both abiotic and biotic components), non-biodegradable in nature and are of great concern [3,4]. Agriculture, development, population increase, urbanization, and industrialization are various causes that are continuous sources of heavy metal pollution in the environment [5–7].

Rapid industrialization has direct and indirect effects on the environment. Industries release harmful gases and toxic waste (solid and liquid) polluting the environment by affecting air, water and soil quality [8–10]. The presence of heavy metals in wastewater can greatly affect different segments of the environment. Sludge is the main source from where heavy metal can seep through water into an aquifer [11,12]. Wastewater containing heavy metals, if used for irrigation, can affect the soil properties and accumulate in soil and plants [13]. In order to understand the pollution level in the aquatic system, it is necessary to assess its distribution in various segments

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of the environment [14].

Sludge also contains microorganisms, organic matter, pathogens, pharmaceuticals, organic and inorganic pollutants depending upon the inputs in effluents (industrial and domestic wastewater) [15]. Zn, Hg, Pb, Cd, Cr, Ni and Cu are the main elements that restrict the use of sludge for land application as it contaminates ground water and soil, which in turn affects human health [16]. As it is a sink and source for different contaminants, it can provide useful information on heavy metals status and ecological risks related to them [17,18].

Over 400 species of plants have been identified as hyper accumulators as they can accumulate high concentrations of heavy metals [19]. The morphology of plants is a contributing factor in accumulating heavy metals [20]. Evaluation of heavy metals in plants is important as it indicates the pollution status of the site or soil and shows the ability of the plant to uptake and accumulate heavy metals [21]. Grasses, which can easily and quickly grow naturally, can be used to monitor pollution [22]. It is necessary to monitor toxic heavy metals in different environmental media to identify associated risk [23].

This study was conducted to find out heavy metals in wastewater and sludge and find out their association and correlation with heavy metals accumulated in the grass. The objective of this study was to investigate heavy metals concentration and distribution in different media; wastewater, sludge and plants of main drains.

2. Materials and method

2.1. Study area/sampling sites

The study was conducted to evaluate the contamination level of effluents of the Hayatabad Industrial estate (HIE), Peshawar. HIE, Peshawar was established in 1963 and covers an area of about 868 acres. It comprises 372 industrial units, of which 242 are functional [24]. Literature showed that industrial effluents from the industrial estate discharged in Budni Nala (main drain) which falls into the Kabul River, thus contaminating water bodies and affecting flora and fauna [25]. In order to monitor the impact of industrial effluents, different environmental media (wastewater, sludge and plants) were collected for analysis. For sampling, five main drains were selected. 4 (sampling sites: 1, 2, 3, 4) of these main drains received effluents (Organic and inorganic wastewater) from a cluster of different industries (steel, marble, wood, match, plastic, glass, rubber, mineral, leather, metal, paper, pharmaceutical and food) and the fifth drain (sampling site: 5) was located far away from industrial area as shown in Fig. 1, was selected as control. Geographical coordinates of main drains are; 33°58′51.9″N 71°25′46.7″E (site 1), 33°59′16.3″N 71°25′33.4″E (site 2), 33°59′40.7″N 71°25′24.4″E (site 3), 33°59′45.6″N 71°26′07.5″E (site 4) and 33°59′49.6″N 71°26′36.6″E (control) respectively.

2.2. Samples collection and preparation

Wastewater samples were collected from 5 points of the main drains in clean plastic bottles, 24 grab samples were collected from each site at a 30 min interval and transferred to the laboratory immediately. In this way, 120 samples were collected and analyzed for different water quality parameters. The samples were analyzed for pH, EC, TSS, TDS and heavy metals according to standard methods. pH was measured using a PHS- 3C pH meter, for EC a digital conductivity meter was used, TDS and TSS were measured using gravimetric method. The wastewater samples were analyzed for heavy metals after filtration [26] using atomic absorption

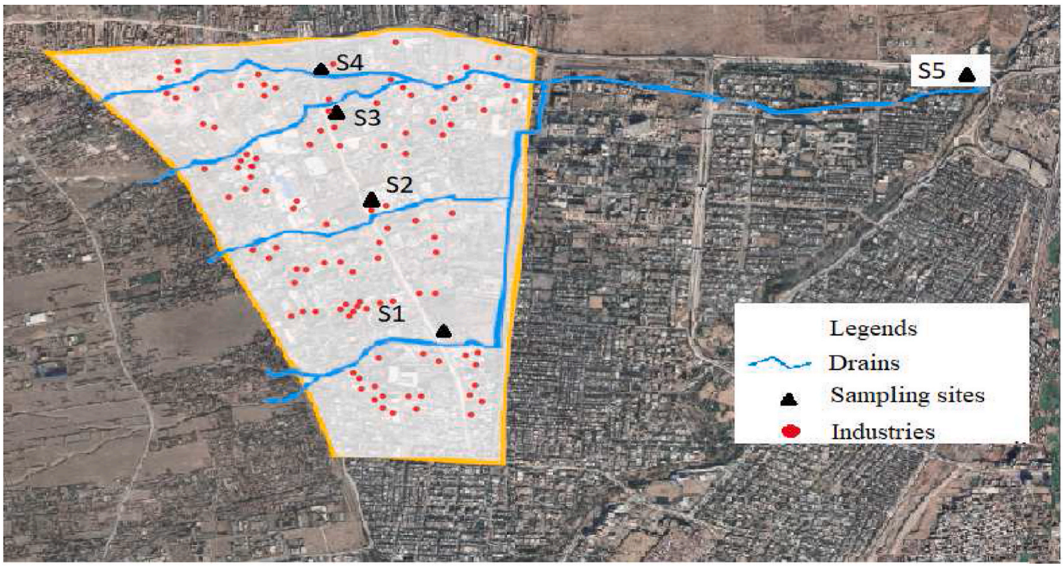

Fig. 1. Map showing drains, sampling areas and distribution of different industries contributing to main drains.

spectrometry (PerkinElmer AAS-700).

Sludge samples (1 kg) from each drain were collected in a clean bag with 3 replications from each site and taken to the laboratory for further analysis. The samples were air dried in the laboratory and sieved (63 μm mesh size). Debris and stone, if present, were removed. The samples were dried further in oven until no change in weight was observed. 1 g of sludge sample was digested using a mixture of 12 ml HNO_3 and 4 ml HCl. The samples were boiled on a hot plate for 2 h and transferred to volumetric flasks and with deionized water volume was made up to 100 ml [27] and analyzed for heavy metals using atomic absorption spectrometry.

Phalaris minor, a grass species, was found commonly at all sample sites of the drain. Samples were collected from all sites and were stored in polyethylene bags. Samples were washed with tap water and rinsed with distilled water and dried in oven at 70 °C for 24 h. The samples were separated into roots and shoot and powdered. 0.5 g sample was transferred to 100 ml Pyrex digestion tube. 10 ml of acid mixture of 1:5 perchloric acid-nitric acid, was added to the sample and placed in a digester block. Starting from 100 °C temperature was gradually increased until dense white fumes appeared, the digestion was completed and, after cooling, deionized water was added to bring the volume to 50 ml. Each batch of samples for analysis also contained reagent blank (no sample) [28]. The plant samples (roots and stem) were analyzed for selected heavy metals (Cd, Cr, Pb and Zn) using atomic absorption spectrometry.

For determination of heavy metals (Cd, Cr, Pb, Pb), standard solutions of the analyte at different concentrations were prepared by diluting stock solution (1000 mg/l) for each element with deionized water. The quality was assessed by examining the linearity and slope of the calibration curve. Each sample was analyzed in triplicate to ensure the quality of data of samples for research. Blank and standard samples were digested and analyzed using the same procedure [26].

In order to ascertain their bioindicator value, the bioconcentration factor was calculated; $BCF = [Metal]_{plant}/[Metal]_{sludge}$, and to find out the ability of plants to remove heavy metals from the surrounding environment and accumulate them.

2.3. Statistical analysis

The correlation was calculated for properties of wastewater, heavy metals in wastewater, sludge and plants using Excel. Pearson's correlation coefficient was used to identify the relationship between metals concentration in plants and sludge, sludge and wastewater using SPSS 17.0 at $p < 0.05$.

3. Results and discussion

3.1. Physicochemical properties of wastewater

3.1.1. pH

pH values for mixed drains of industrial effluents and main drains ranged from 6.44 to 7.31. The lowest value was reported for site 1 and the highest was recorded for the control site (Table 1). In comparison with Pakistan National Environmental Quality Standards (Pak-NEQS) [29], the results of all 5 sites were found within permissible limits.

pH is the measure of acidity and alkalinity of water. It is an important factor as aquatic life and chemical reactions are affected by any change in it. It determines the quality of water and small changes in it make it unfit for use. pH at the control site (main drain) was reported high as compared to other sites. The results of this study are in contrast to a previous study conducted on combined drains of different industries of Hattar industrial estate, which revealed that pH of effluents was found between 4.6 and 10.9 (acidic to alkaline) and some of them exceeded permissible limits. This range was observed due to different sampling periods [30]. [31] reported similar results of pH for wastewater effluents of different industries. Toxicity and solubility of many substances depend on pH. At low pH many substances are readily available for adsorption as the solubility of insoluble substances increases [30,31].

3.1.2. Electrical conductivity (EC)

The EC for main drains ranged between 1000 and 1800 μS/m. The lowest value was found for the main drain of site 3 with a mean value of 1000 μS/m and the highest for site 4 with a mean value of 1800 μS/m (Table 1).

Electrical conductivity is the ability of water to conduct electric current which shows the concentration of ions and their mobility. Inorganic compounds tend to be good conductors of electricity as compared to organic compounds. Highest EC at site 4 is due to the combined effluents of all industries, resulting in an increase in salt concentration. The ion concentration is the result of dissolved solids

Table 1
Physicochemical parameters of 5 main effluent drains of Hayatabad Industrial Estate, Peshawar.

Sampling sites	pH			EC			TSS			TDS		
	Min	Max	AVG	Min	Max	AVG	Min	Max	AVG	Min	Max	AVG
1	6	6.67	6.44	1600	1700	1627	38	510	271	785	846	813
2	6.01	7.49	6.64	1400	1500	1480	54	1664	469	703	980	819
3	6.66	7.1	6.92	1000	1000	1000	18	2946	901	486	660	560
4	6.53	6.87	6.72	1700	1900	1800	688	1524	1089	840	1183	942
Control	7.13	7.4	7.31	1200	1200	1200	310	394	347	574	799	673
Pak-NEQS[a]	6–10			–			150 mg/L			3500 mg/L		

[a] Values represent maximum permissible limits of parameters according to Pak-NEQS (Pakistan National Environmental Quality Standards).

mainly and at control site EC reduced to 1200 μS/m which might be due to the ions precipitation and settling. The results are supported by Ref. [32] work which showed a decrease in EC with the increase in distance from industries and with the change in total dissolved solids (TDS).

3.1.3. Total suspended solids (TSS)

Total suspended solids for the effluents ranged from 271 to 1089 mg/L. The lowest value was observed for main drain of site 1 (271 mg/L) and the highest value for main drain of site 4 (1089 mg/L) (Table 1).

When compared with [29] Pak-NEQ'S, the effluents of all 5 sites exceeded permissible limits. Highest TSS for site 4 may be due to collective industrial effluent. The highest values reported for site 3 were due to the effluents from marble industries as they carry solid particles after being used during the crushing process. Similar results were reported by Refs. [30,33] for high levels of TSS in effluents of different industries.

3.1.4. Total dissolved solids (TDS)

TDS of effluent for 5 different sites ranged between 560 and 942 mg/L. The lowest value was recorded for the main drain of site 3 and the highest for main drain of site 4 (Table 1). It is evident from the results that TDS for all sites were within limits.

Total dissolved solid is the measure of salinity in water. A high concentration of TDS changes its density, decreases the solubility of gases, affects aquatic organisms and, when discharged into water used for irrigation purpose, may cause salinity. As TDS values were in compliance with [29] Pak-NEQ'S, site 4 was high in TDS content. That may be due to the collective effluent of all contributing industries which resulted in high ion concentration in water. After a careful look, it seems that the steel re-rolling mill and the match industry are the main contributing industries. In the steel re-rolling mill, water is used to remove dust to treat blast furnace gas during smelting, as well as a cooling agent, while in the match industry, water is used during chemical mixing and washing of waste [24,34,35].

EC and TDS were significantly correlated (r = 0.990) which shows that TDS increased with increase in EC (Table 2). EC and TDS give values for ionic load and contaminants in water. TDS is mineral salt dissolved in water and EC is the ability of water to conduct electricity which depends upon the ionic strength of water thus, EC is a function of salt concentration. pH and EC (r = - 0.642), pH and TDS (r = - 0.573) showed moderate but negative correlation. An increase in one parameter resulted in a decrease in the other. pH depends on H+ and OH- ions concentration only, which comprises a small part of conductivity. There is a direct relation between EC and TDS, an increase in TDS results in an increase in EC. The end products of organic and inorganic compounds used in industries can affect pH, EC and TDS of effluents [36]. Metals, Na^{+}, Ca^{2+}, SO_4^{2-}, Mg^{2+}, HCO^{3-} and K^{+} are contributing factors to high EC and TDS [37].

3.2. Heavy metals concentration in wastewater

The average concentrations of Cd, Cr, Pb and Zn for each site are given in Table 3. Cd concentration in wastewater samples ranged from 8 to 38 μg/L. The lowest value of Cd was observed for the control site and the highest for site 2 industries. The level of Cr in wastewater samples ranged from 40 to 254 μg/L. The lowest value of Cr was recorded for the control site and the highest for site 2. Pb ranged from 46 to 96 μg/L in wastewater. The lowest value for Pb was noted for control and the highest for site 2. Concentration of Zn ranged between 43 and 1355 μg/L. The lowest was recorded for control and higher for Site 4 (Table 3). Site 5 which receives wastewater from streams carrying contaminated water, showed low metal concentration is because of dilution effect. Dilution plays an important role in reducing/dispersing contaminants, due to mixing of these effluents with a large volume of water. At this point, the North nullah a natural transport system, which crosses the industrial estate. It helps in taking effluent away from industrial estate to Budhni nullah which joins Shah Alam and then eventually into Kabul river.

By comparing the results with [29] Pak-NEQS, wastewater samples were found within permissible limits for Cd, Cr, Pb and Zn. In comparison with FAO limits [38] for the use of wastewater for crop production, heavy metals concentration was within permissible limits except Cd which was recorded high for all sites and Cr for site 2. Contamination level in the 4 main industrial effluent drains and Cd use source can be attributed to steel, plastic, marble and metal plating industries. Average Cd concentration is recorded lowest as compared to Cr, Pb and Zn for all sites. Cr sources at site 1 can be attributed to steel, wood and food preservative industries. The order of heavy metals for all sites were different, which was due to the different industries. Metals concentration was lowest at control site, which may be due to high pH, as heavy metals at high pH are removed from water and adsorb to sediments. In this study, the control site showed the highest pH with an average value of 7.31 as compared to other 4 sites. Higher pH can remove heavy metals from water by precipitation process resulting in hydroxide formation and adsorb to sediments [39].

The results of this study are in line with the results of previous study [33] conducted on wastewater of marble, match, steel,

Table 2
Correlation of physicochemical parameters in wastewater of HIE, Peshawar.

	pH	EC	TSS	TDS
pH	1			
EC	−0.642	1		
TSS	0.015	0.099	1	
TDS	−0.573	**0.990**	0.120	1

Table 3
Heavy metal concentration in effluents of 5 main drains of Hayatabad Industrial Estate, Peshawar.

Sites	Cd μg/L			Cr μg/L			Pb μg/L			Zn μg/L		
	Min	Max	Avg	Min	Max	Avg	Min	Max	Avg	Min	Max	Avg
1	2	69	16	3	91	45	5	185	61	3	106	47
2	5	77	38	62	541	254	24	187	96	84	911	436
3	4	42	16	2	99	48	14	111	64	19	186	95
4	2	17	8	13	96	51	22	177	95	843	1904	1355
Control	1	19	8	15	71	40	11	99	46	12	84	43
WHO/FAO limits (2007) [38]	10 μg/L			100 μg/L			5000 μg/L			2000 μg/L		
Pak-NEQS[a] [29]	100 μg/L			1000 μg/L			500 μg/L			5000 μg/L		

[a] Values represent maximum permissible limits of parameters according to Pak-NEQS (Pakistan National Environmental Quality Standards).

pharmaceutical industries and main drain of HIE, Peshawar that showed Cd, Cr and Zn concentration in effluents of respective industries were within permissible limits when compared to Ref. [29] Pak-NEQS except for Pb in effluents of marble, match, steel and main drain exceeded limits [31]. reported high concentration of metals Cd, Cr, Pb and Zn in effluents of the match, paper mill, marble, woolen mill and glass industries when compared with [29] Pak-NEQS which is contrary to this study. Cd and Cr showed significant positive correlation ($r = 0.946$, $p < 0.05$) (Table 4). Similarly a positive correlation between Cd with Pb, Cr with Pb, and Pb with Zn suggest that these metals might have originated from the same source [40].

3.3. Heavy metals concentration in sludge

The average concentrations of heavy metals in sludge samples of the five sites are presented in Table 5. Cd concentration was found in the range of 27–1101 μg/kg, the highest concentration was found at site 2 (1101 μg/kg). Cr concentration was found in the range of 622–4120 μg/kg; the highest concentration was found at site 1. Pb concentration in sludge ranged from 168 to 4901 μg/kg; the highest concentration of Pb (4901 μg/kg) was found at site 3. Zn concentration ranged from 1084 to 3337 μg/kg; the highest concentration was found at site 1 (Table 5). The results showed that metal concentration varied at each site and was recorded higher for site 1, 2, 3 and 4 which are main drains which directly receive industrial effluents as compared to control. Metals concentration in the sludge was compared with [41] permissible limits for metals in sludge and the values were within limits.

The concentrations of heavy metals recorded in sludge samples were higher than wastewater, as heavy metals from water accumulate in sludge [42]. The reason might be adsorption of heavy metals to particles suspended in water which settle down and become a secondary source of contamination [43]. The order of heavy metals in sludge was not similar to that of wastewater. pH affects the heavy metals concentration in sludge. As pH increases, heavy metals precipitates due to hydroxide production and adsorbs to sediment [39]. Under slightly acidic pH, soluble Zn is found high and is mobile and bioavailable as compared to basic pH [44]. In this study, pH of wastewater ranged from 6 to 7.49, so the level of Zn was found high as compared to Pb, Cr and Cd in sludge in this study. As a by-product of wastewater treatment plant, the quality of sludge depends upon the chemical properties of wastewater and the treatment process involved [45]. Table 6, shows weak and moderate correlation of heavy metals in sludge samples. It can be assumed that moderate correlation might be due to metals originating from the same source or similar accumulation behaviour in sludge and the negative correlation may reflect similar behavior and competitive adsorption [43]. Similar results were also reported by Ref. [40] in sludge samples.

3.4. Heavy metals concentration in plants

The concentrations of heavy metals in different parts of the plants collected from study area are given in Table 7. In plants Cd, Cr, Pb and Zn concentrations ranged from 0 to 19 μg/kg, 11–3021 μg/kg, 1–2813 μg/kg and 72–2733 μg/kg. Similar results were reported in a previous study [46]; an increase in heavy metals concentration (Pb. Cd, Co) was recorded in *Phalaris minor* with an increase in soil concentration and was lower as compared to *P. monospensis*.

In this study, roots showed a higher concentration of heavy metals as compared to shoots. This is because roots act as a barrier for translocation of metals in order to protect the shoots from the effects of metals [47]. Same plant species having different levels of heavy metals at different sites show that plant's uptake and transport depend upon the level of heavy metals in the soil and the ability of plants to accumulate such heavy metals. The results of this study are in line with previous studies reporting high retention of heavy metals in roots [48,49]. In shoots Cr and Zn showed significant positive correlation (0.810) (Table 8), this could be due to the same

Table 4
Correlation of heavy metals in wastewater of HIE, Peshawar.

	Cd	Cr	Pb	Zn
Cd	1			
Cr	**0.946**	1		
Pb	0.504	0.627	1	
Zn	−0.144	0.073	0.773	1

Table 5
Heavy metals concentration μg/kg in sludge sample of HIE, Peshawar.

Sampling sites	Cd μg/kg	Cr μg/kg	Pb μg/kg	Zn μg/kg
1	1006	622	2788	3337
2	1101	2974	1132	2015
3	55	2101	4901	2029
4	1013	4120	1392	1621
Control	27	1045	168	1084
USEPA, (2018) [39]	39000 μg/kg	1200000 μg/kg	300000 μg/kg	2800000 μg/kg

Table 6
Correlation of heavy metals in sludge samples.

	Cd	Cr	Pb	Zn
Cd	1			
Cr	0.404	1		
Pb	−0.227	−0.119	1	
Zn	0.493	−0.373	0.495	1

Table 7
Heavy metals concentration μg/kg in plants samples of HIE, Peshawar.

Sampling sites	Cd μg/kg		Cr μg/kg		Pb μg/kg		Zn μg/kg	
	Root	Shoot	Root	Shoot	Root	Shoot	Root	Shoot
1	19	3	3021	1015	215	7	2308	2002
2	7	1	2611	1038	10	12	1920	1991
3	1	0	1042	11	2813	1109	968	72
4	11	5	834	1010	1015	781	2733	1519
Control	3	0	201	702	399	1	2501	36
WHO/FAO Limits (2007) [36]	200 μg/kg		5000 μg/kg		5000 μg/kg		60000 μg/kg	

path for transport and requirement of essential nutrients for plants [50]. In roots Cr and Pb (−0.740) and Zn with Pb (-0.722) showed negative correlation which could be due to competition between heavy metals (Cd, Pb and Zn) of similar chemical properties for uptake [51]. Cd showed weak correlation with Cr, Pb and Zn in both root and shoot. Different capabilities of plants to uptake heavy metals might have resulted in weak correlation between metals in plants [52]. Metals distribution in plants mainly depends upon plant species, water transport, availability and form [53]. Translocation of metals and compartmentalization in plant tissues are the reasons for variations in concentration of metals and stem acts as a pathway for transfer of metals thus low metals concentration is recorded in shoots [54].

Heavy metals can easily accumulate in the surrounding environment and are of concern due to their toxicity [55]. Weed plants have been used to remediate heavy metal contaminated sites due to high biomass, growth rate and tolerance to toxic pollutants [56]. Studies conducted on five different plant species (*L. spartum, G. decander, A. serratuloides, E. Glaucophyllum, H. Kahirikum*) colonized closely to the cement industry showed higher translocation and bioaccumulation of heavy metals in perennial against annual species and was reported that plants could be potential indicator of heavy metals. *L. spartum, G. decander, A. serratuloides* were identified as pollution indicators [57]. The identification of plants growing at sites contaminated with heavy metals, can provide information about the mechanism of tolerance to heavy metals and their accumulation in plants [58]. On one hand, heavy metals are accumulated in plants specifically in their roots and their correlation with heavy metals in substrate may represent the level of contamination, but on the

Table 8
Correlation matrix of heavy metals in roots of plants samples.

	Cd	Cr	Pb	Zn
Cd	1			
Cr	0.508	1		
Pb	−0.527	−0.740	1	
Zn	0.520	0.169	−0.722	1
Correlation of heavy metals in shoot of plants samples				
	Cd	***Cr***	***Pb***	***Zn***
Cd	1			
Cr	0.614	1		
Pb	0.109	−0.672	1	
Zn	0.634	**0.810**	−0.402	1

other hand, these accumulated heavy metals upon subsequent release into the environment during decomposition results in recycling of heavy metals in the environment. Such a pathway can have an important effect on the level of toxic heavy metals in the surrounding environment [59].

3.5. Bioconcentration factors (BCF) for metals in plants

Table 9 showed bioconcentration factors, the ratio of metal concentration in plants to that in sludge. BCF greater than 1 shows the ability of plants to accumulate metals from the environment and shows the ability for accumulation in tissues. As Cd in some plant samples was below detectable limit BCF was not available. The highest BCF was observed for Cr (4.8) in roots. BCF greater than 1 was observed for Cr (root, shoot), Pb (root) and Zn (root), which showed high phytoremediation potential and Cd showed the lowest < 1 BCF. BCF for Cd was <1, which indicated that this species cannot be used as bioindicator for Cd but BCF >1 was observed for Cr, Pb and Zn, which showed that this plant species can be used as bioindicator for Cr, Pb and Zn. The results of this study revealed that *Phalaris minor* can be used for biomonitoring purposes. In previous studies, greater BCF values were also reported for Pb and Zn in other grass species (*C. pennisetiformis, C. dactylon,* and *B. reptans*) which showed the phytostabilizing potential of grasses and availability of metals to plants [60].

3.6. Correlation of heavy metals between plants (root, shoot) and sludge

The high correlation of heavy metals between sludge and roots were observed for Cd (0.754) and for Pb (0.793) (Table 10), which indicates that increase in Cd and Pb in sludge resulted in an increase in plant roots. The strong correlation of plant tissues with that of sludge indicated heavy metal bioavailability [61]. Zn and Pb showed a highly negative correlation (-0.802) which shows that with the increasing Zn in sludge Pb uptake in plant roots, reduced. The rest of the elements showed non-significant and weak correlation. The reason for the weak correlation between plants and sludge might be due to the fact that correlation was calculated with total metal concentrations rather than the bioavailable fraction in sludge [62]. Metal concentration in plants is mainly affected by their availability in sludge and high concentration of metals in sludge [21]. Metal accumulation in plants generally depends upon plant species, heavy metal properties, uptake, translocation of metals, pH of soil, metals concentration in soil and organic matter [21,63]. Similar results for weak correlation in soil and plants (grass) were also reported by Refs. [22,64]. [65] also reported the absence of correlation of metals in plants with sediment.

Correlation of heavy metals in sludge and shoots showed significantly strong and positive correlation was found between Cd and Zn ($r = 0.984^{**}$, $p < 0.01$). Cd showed strong and positive correlation with Cr (0.82) and weak and negative correlation with Pb (-0.303) (Table 11). The significantly strong and positive correlation between Cd and Zn shows that Cd concentration in sludge influenced Zn uptake in plants. The strong correlation of heavy metals in soil with plants shows availability of that metal in soil and its transport and accumulation in shoot also depend on being essential for it [66]. The present study showed that most of the heavy metals in sludge showed positive correlation with those in plants except Pb. Positive correlations indicate the effect of metals in sludge on metals absorbed by plants. Such correlation can be supported by several other studies in which the total quantity of metals in sludge and soil correlated with metals in plants [67,68].

3.7. Correlation of heavy metals between sludge and wastewater

Table 12 shows the correlation of heavy metals in sludge with wastewater, which revealed a significantly strong and positive correlation between Cr and Pb ($r = 0.879$, $p = 0.05$), and Cr and Zn ($r = 0.895$, $p < 0.05$) and high correlation of Cd with Pb in wastewater ($r = 0.745$, $p > 0.05$). The results revealed that increase in one element corresponded to increase in other, which indicated close association of heavy metals in sludge and wastewater. Negative correlation was also observed for Pb with Cr, Pb and Zn in wastewater. The results of correlation of heavy metals suggest that a positive correlation between different metals might be due to the fact that these metals either have similar pollution levels, transport and common sources (industries) or a single source from which these heavy metals originated [69]. Industries such as steel mills, mechanical processing, dyeing are the main sources of heavy metals (Cd, Cr, Pb and Zn). The results similar to this study were also reported by Ref. [70].

Table 9
Bioconcentration factors for heavy metals.

Sampling sites	Cd		Cr		Pb		Zn	
	Root	Shoot	Root	Shoot	Root	Shoot	Root	Shoot
1	0.018	0.002	4.8	1.631	0.077	0.002	0.692	0.599
2	0.006	0.009	0.877	0.34	0.008	0.01	0.95	0.988
3	0.018	–	0.49	0.005	0.573	0.226	0.477	0.035
4	0.01	0.004	0.202	0.245	0.729	0.561	1.685	0.937
Control	0.111	–	0.923	0.671	2.375	0.005	2.307	0.033

Table 10
Pearson's correlation matrix between heavy metals of sludge and roots of plants of HIE.

Heavy metals in sludge		Heavy metals in plant roots			
		Cd	Cr	Pb	Zn
Cd	Pearson Correlation	0.754	0.37	−0.572	0.425
	Sig. (2-tailed)	0.141	0.54	0.313	0.476
	N	5	5	5	5
Cr	Pearson Correlation	−0.144	−0.608	0.154	0.1
	Sig. (2-tailed)	0.817	0.277	0.804	0.873
	N	5	5	5	5
Pb	Pearson Correlation	−0.067	−0.253	0.793	**−0.802**
	Sig. (2-tailed)	0.915	0.681	0.109	0.103
	N	5	5	5	5
Zn	Pearson Correlation	0.752	0.562	−0.121	−0.158
	Sig. (2-tailed)	0.142	0.324	0.847	0.8
	N	5	5	5	5

* Correlation is significant at the 0.05 level (2-tailed).

Table 11
Pearson's correlation matrix between heavy metals of sludge and shoots of plants of HIE.

Heavy metals in sludge		Heavy metals in plant shoots			
		Cd	Cr	Pb	Zn
Cd	Pearson Correlation	0.719	0.82	−0.303	**0.984****
	Sig. (2-tailed)	0.171	0.09	0.62	0.002
	N	5	5	5	5
Cr	Pearson Correlation	0.477	0.173	0.48	0.248
	Sig. (2-tailed)	0.417	0.78	0.414	0.688
	N	5	5	5	5
Pb	Pearson Correlation	−0.14	−0.701	0.669	−0.186
	Sig. (2-tailed)	0.822	0.187	0.217	0.765
	N	5	5	5	5
Zn	Pearson Correlation	0.274	0.193	−0.164	0.604
	Sig. (2-tailed)	0.656	0.755	0.792	0.281
	N	5	5	5	5

** Correlation is significant at the 0.01 level (2-tailed).

Table 12
Pearson's correlation matrix between heavy metals in sludge and wastewater of HIE.

Heavy metals in sludge		Heavy metals in wastewater			
		Cd	Cr	Pb	Zn
Cd	Pearson Correlation	0.447	0.489	0.745	0.521
	Sig. (2-tailed)	0.45	0.404	0.148	0.368
	N	5	5	5	5
Cr	Pearson Correlation	0.158	0.348	**.879***	**.895***
	Sig. (2-tailed)	0.8	0.566	0.05	0.04
	N	5	5	5	5
Pb	Pearson Correlation	0	−0.268	−0.118	−0.272
	Sig. (2-tailed)	1	0.663	0.85	0.658
	N	5	5	5	5
Zn	Pearson Correlation	0.259	0.006	0.007	−0.276
	Sig. (2-tailed)	0.674	0.992	0.992	0.653
	N	5	5	5	5

*. Correlation is significant at the 0.05 level (2-tailed).

4. Conclusion

Despite the fact that heavy metals in wastewater meet the Pak-NEQS but can still pose a threat to human health and the environment due to Cd and Cr excess, when compared with FAO limits for irrigation of crops. Heavy metals were differently distributed among studied compartments; sludge > plants > wastewater. For wastewater and sludge, heavy metals concentration was highest at sites within industrial estate and compared to control. In plants, the order of metals was different for each site as compared to control, and most metals accumulated highest in roots as compared to shoots. Positive correlation of heavy metals in sludge with plant roots and shoots, revealed that accumulation of heavy metals in plants was through sludge. The BCF for Cr, Pb and Zn was greater than 1 except for Cd, and can be used as bioindicator for Cr, Pb and Zn. The findings in this study revealed that the total metals concentration in the grass *Phalaris minor* can give information about pollution level. The grass is also useful to find out bioavailability, toxicity and mobility of metals and as an indicator of potential risk. Thus, monitoring the impacts of industrial activities is important to find out its trend and pattern of distribution in different compartments of the environment. It is important to note that the present study focused specifically on analysis and distribution of Cd, Cr, Pb, and Zn in different compartments of the environment and their accumulation in plants. The research did not include other heavy metals. The inclusion of additional heavy metals in future research would provide valuable insights into their respective uptake mechanisms in plants and their relationship with the aforementioned elements.

Author contribution statement

Sara Nawaz Khan; Dr Mohammad Nafees; Dr Muhammad Imtiaz: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.

Data availability statement

Data included in article/supplementary material/referenced in article.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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