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Source and Ecological Risk Assessment of Potentially Toxic Metals in Urban Riverine Sediments Using Multivariate Analytical and Statistical Tools

Xiaojun Zheng [1,2,†], Abdul Rehman [1,†], Shan Zhong [3,*], Shah Faisal [4], Muhammad Mahroz Hussain [5], Syeda Urooj Fatima [6] and Daolin Du [7,*]

1 School of Environment and Safety Engineering, Jiangsu University, Zhenjiang 212013, China; xjzheng@ujs.edu.cn (X.Z.); 1000006602@ujs.edu.cn (A.R.)
2 Jiangsu Collaborative Innovation Center of Technology and Material of Water Treatment, Suzhou University of Science and Technology, Suzhou 215009, China
3 School of Energy and Power Engineering, Jiangsu University, Zhenjiang 212013, China
4 Department of Environmental Engineering, School of Architecture and Civil Engineering, Chengdu University, Chengdu 610106, China; shahfaisal@cdu.edu.cn
5 College of Environment Science and Engineering, Yangzhou University, Yangzhou 225009, China; 008785@yzu.edu.cn
6 Institute of Environmental Studies, University of Karachi, Karachi 75270, Pakistan; syedauroojfatima.ku@gmail.com
7 Jingjiang College, Institute of Environment and Ecology, School of Environment and Safety Engineering, School of Emergency Management, School of Agricultural Engineering, Jiangsu University, Zhenjiang 212013, China
* Correspondence: szhong@ujs.edu.cn (S.Z.); ddl@ujs.edu.cn (D.D.)
† These authors contributed equally to this work.

Abstract: Multivariate and statistical tool advancements help to assess potential pollution threats, their geochemical distribution, and the competition between natural and anthropogenic influences, particularly on sediment contamination with potentially toxic metals (PTMs). For this, riverine sediments from 25 locations along urban banksides of the River Ravi, Pakistan, were collected and analyzed to explore the distribution, pollution, ecological, and toxicity risk indices of PTMs like Al, As, Cd, Co, Cr, Cu, Fe, Hg, Mn, Ni, Pb, Sb, Sn, Sr, V, and Zn using Inductively Coupled Plasma–Optical Emission Spectrometry (ICP-OES) technique. Additionally, techniques such as X-ray Diffraction (XRD) and Scanning Electron Microscopy–Energy Dispersive X-ray Spectroscopy (SEM-EDS) were employed to investigate the mineralogical and morphological aspects. The results indicated that mean concentrations (mg kg^{-1}) of Cd (2.37), Cr (128), Hg (16.6), Pb (26.6), and Sb (2.44) were significantly higher than reference values given for upper continental crust (UCC) and world soil average (WSA), posing potential threats. Furthermore, the geochemical pollution indices showed that sediments were moderately polluted with Cd (I_{geo} = 2.37, EF = 12.1, and CF = 7.89) and extremely polluted with Hg (I_{geo} = 4.54, EF = 63.2, and CF = 41.41). Ecological and toxicity risks were calculated to be extremely high, using respective models, predominantly due to Hg (Er_i = 1656 and ITRI = 91.6). SEM-EDS illustrated the small extent of anthropogenic particles having predominant concentrations of Zn, Fe, Pb, and Sr. Multivariate statistical analyses revealed significant associations between the concentrations of PTMs and the sampling locations, highlighting the anthropogenic contributions linked to local land-use characteristics. The present study concludes that River Ravi sediments exhibit moderate levels of Cd and extreme pollution by Hg, both of which contribute highly to extreme ecological and toxicity risks, influenced by both natural and anthropogenic contributions.

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1. Introduction

Riverine sediments play a fundamental role in the preservation of the aquatic and terrestrial ecosystems, affecting habitat, soil, and aquatic life. Any physical, chemical, or biological disturbances within these sediments can significantly impact various components attributed to the riverine environment [1]. In the past few decades, anthropogenic activities such as the discharge of industrial effluents, intensified agricultural practices, improper waste management, and urban expansion have posed serious threats to the riverine ecosystem, thus contributing to the contamination of these sediments with potentially toxic metals (PTMs) [2,3]. These PTMs have a noxious nature, long half-life, and bioaccumulation/translocation attributes in aquatic sediments that can adversely affect aquatic and terrestrial ecosystems, posing a threat to the food chain and, ultimately, humans [4].

The distribution of PTMs within sedimentary deposits offers researchers valuable insights into the anthropogenic effects on aquatic ecosystems, which aids in evaluating the risks linked to human activities, as these can serve as both carriers and sources of contamination [5]. Contamination of riverine sediments with PTMs originated from various sources, either via human or natural processes, targeting low-income countries through less-organized management practices [6].

The riverine system of Pakistan primarily depends on the Indus River network, which is the lifeline of Pakistan's economy and has deteriorated significantly, due to the discharge of untreated industrial and domestic wastewater, solid waste disposal, and agricultural runoff [7]. However, studies suggested that, along with the anthropic factors, the concentrations of specific PTMs may be conclusively associated with geogenic processes and natural sources [1]. However, to overcome potential contamination risk, its source identification and indexical approaches have been reported as valuable tools for both aquatic and soil-sediment contaminations. Therefore, source apportionment of contaminants had been employed in previous studies through multivariate statistical techniques [8] and integration of geochemical data for compositional analyses [9].

Furthermore, combining chemical, toxicological, and ecological data allows for a comprehensive evaluation of the impact of PTMs pollution, as reported in studies conducted on sediments of various aquatic ecosystems of the world, including the River Ravi in Pakistan and the Beas River in India [10], the Bahmanshir River, Iran [11], Lake Qarun [12], the Nile River, Egypt [13], Kaptai Lake, Bangladesh [14], Caohai Wetland [15], Xiaoqing River Basin, China [16], estuarine sediments of the Southeastern Black Sea, Turkey [17], and the northern Amazon region of Ecuador [18]. However, several other studies reported elevated PTM pollution in rivers and lakes of Pakistan, accounting for the River Indus [19–21], Kabul, Sutlej, Ravi, Jhelum, and Chenab rivers [3], Uchalli lake Ramsar site [4], Keenjhar Lake [22], and others [23–25]. Nevertheless, the information on the River Ravi regarding PTM pollution status is lacking, despite the continuous anthropogenic input with the rapid increase in urbanization.

Combining geochemical, ecological, and multivariate statistical tools can elucidate the potential threat of the PTMs under varying conditions. So, this study aimed to provide useful insight into the distribution of PTMs pollution in riverine sediments of the River Ravi, Pakistan, which covers a significant area for both agricultural and commercial use. The current research work looks at the role of riverine sediments as indicators for evaluating the level of PTM contamination and in identifying their sources. The objectives of the present study were to (1) evaluate the status of distribution and concentration levels of selected PTMs in riverine sediments in both agricultural and commercial zones; (2) characterize the pollution levels and ecological risk assessment of PTMs through the calculation of multiple risk indices; and (3) the characterization of the riverine sediments using XRD for mineralogical aspects and SEM-EDS for morphological aspects.

2. Materials and Methods

2.1. Study Area Description and Sampling

The Ravi River serves as a significant transboundary river shared by India and Pakistan. It holds crucial importance, as it constitutes a vital component of the Indus River basin and serves as the headwater of the Indus Basin system. It nourishes diverse landscapes, where lush agricultural fields meet bustling commercial zones, particularly surrounded by the study area. However, this balance has been threatened by PTM pollution with agricultural runoff and industrial discharges, and, specifically, the influence of chemical processing plants contributing significantly to sediment contamination [3,26]. The study area mainly focused on the 75 km stretch of the River Ravi, which flows along the boundaries touching the areas of three districts, including Lahore, Kasur, and Nankana Sahib.

Sediment samples were collected from 25 location points (details are given in Table S1) along the banksides of the river (Figure 1) in August–September 2022. The locations of the sample collection points were decided based on local land-use characteristics observed on the site, e.g., roadside traffic activities, industrial activities, residential populated areas, agricultural activities, and forest reserve areas. A steel core sampler (3-inch diameter) was used to collect the sediment samples from a depth down to 0–15 cm. The collected sediment samples were shifted into clean polyethylene zip-locked bags and stored in the ice box. Samples were screened and sieved under controlled conditions to avoid any external contamination.

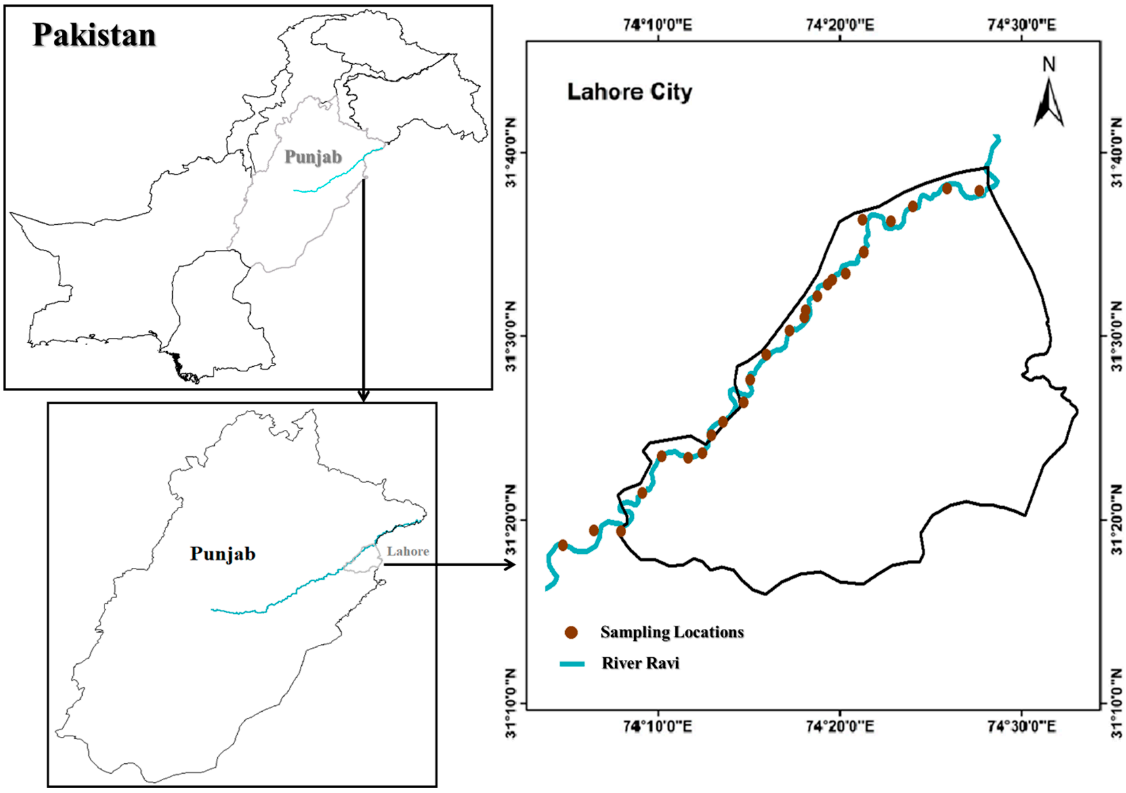

Figure 1. Location of the sampling sites across the River Ravi. Details of the sampling area, geographical coordinates, and land-use type attributed to each location are provided in Table S1.

2.2. Organic Matter and Grain Size Analyses

The loss-on-ignition method was used to determine the total organic matter (TOM). For this, sediments were dried at 80 °C for two days and then combusted in a muffle furnace at 550 °C for 6 h, and TOM was quantified using the equation

$$TOM(\%) = \left({}^{(iw - fw)}/_{iw} \right) \times 100 \quad (1)$$

where *iw* and *fw* represent the initial and final weight of the sediments, respectively, before and after the combustion in the furnace. The calculation showed that the mean value of TOM (%) was 4.26 ± 1.52, ranging from 1.04% to 7.43%. However, a laser diffraction particle-size analyzer was employed to determine the grain size distribution of sediments, which were classified as sand (2000–63 µm), silt (63–4 µm), and clay (<4 µm). The results showed that sediments had mean values (%) for sand, silt, and clay of 27.64 ± 4.62, 50.81 ± 2.96, and 21.55 ± 6.02, respectively.

2.3. Instrumental Analyses

2.3.1. ICP-OES Analysis

All the samples were again dried at 80 °C for 48 h before milling and then sieving with 63 µm nylon mesh. A total of 0.5 g of each sieved sediment sample was taken for digestion, with the help of aqua regia solution. The detailed procedure for acid digestion of sediment samples was adopted, the same as used in our previous study [27]. However, the digestion procedure to prepare samples for instrumental analyses was conducted in the laboratory affiliated with the School of Environment and Safety Engineering of Jiangsu University. Digested sediment samples were analyzed using state-of-the-art ICP-OES. For quality assurance, three reagent blanks, triplicate sample analysis, and certified reference materials were used. The determined recoveries for total PTM concentrations in NIST SRM 8704 and certified marine sediment GBW07314 were found to be within the range of 83.6% to 106.2%. The reliability of the analytical procedure was further tested by analyzing standard solutions after every tenth sample, to ensure precision. The relative standard deviation was maintained at 2.5%.

2.3.2. XRD and SEM Analyses

Representative sediment samples were selected based on ICP-OES results (relatively high PTM carrying) for XRD and SEM-EDS analyses, to study the mineralogy and physical characteristics, respectively. For XRD, the prerequisites of sample preparations were adopted from [28]. However, the XRD (Rigaku, Tokyo, Japan) (TTR-III) conditions were followed as mentioned in [29], to take diffractogram patterns for mineral identification. The scans were taken for sediment samples from the 10°–70° range, and the peaks were identified with corresponding minerals using the X'pert HighScore Plus analyzer (version 3.0e). For SEM-EDS, the samples were mounted on SEM (ESEM, FEI Quanta 250 FEG, Thermo Fisher, Waltham, MA, USA) after the dispersion of an adequate amount on carbon tape, to investigate the anthropogenic influence on the riverine sediments. Moreover, the EDS mode helped to give semi-quantitative results to support the PTMs occurrence, due to anthropogenic particles in the sediments.

2.4. Multivariate Statistical Analysis

The Pearson correlation coefficient (PCC) was calculated to identify significant associations between the potentially toxic metals (PTMs) in sediment samples [15]. Principal component analysis (PCA) was used to assess the concentration dynamics of PTMs by reducing data dimensionality and identifying key factors influencing the environmental characteristics [30]. Cluster analysis (CA) using a hierarchical dendrogram with the Q-mode approach was performed to group similar observations based on associations among sampling locations, to identify potential sources of PTMs. All statistical analyses were performed using OriginPro Lab 2022.

2.5. Assessment of PTM Pollution

Several indices, including the geo-accumulation index (I_{geo}), enrichment factor (EF), pollution load index (PLI), ecological risk index, integrated toxic risk index (ITRI), Nemerow pollution index (NPI), and modified hazard quotient (mHQ) were employed to estimate the pollution levels of PTMs in sediments. Each of these indices has its unique characteristics and limitations, providing different perspectives on pollution status. Therefore, to facilitate the comparison among applicability and interpretation of these pollution indices, they have been summarized in Table S2. The calculation methods for each of these pollution indices are outlined below.

2.5.1. Geo-Accumulation Index (I_{geo})

The accumulation of PTMs was assessed by the geo-accumulation index (I_{geo}) by following [31]. The geo-accumulation index helps differentiate between natural and anthropogenic concentrations of PTMs, providing an indication of the potential extent of environmental contamination. The degree of pseudo-PTM contamination was calculated as

$$I_{geo} = \log_2(C_i/1.5B_i) \quad (2)$$

where C_i is the concentration of the ith PTM, 1.5 is the constant value used to account for possible variations in reference background PTM concentrations due to lithogenic effects, and B_i is the background concentration of the respective PTM.

2.5.2. Enrichment Factor (EF)

The enrichment factor (EF) explains the enrichment level of PTMs pertaining to the influence of anthropogenic and/or natural sources [32,33]. A geochemically stable element is involved in the calculation of EF (i.e., Al, Fe, Sc, or Ti), and in the present study, Fe was used as a reference element. The formula for EF calculations is given as

$$EF = \frac{(C_i/C_{Fe})_s}{(C_i/C_{Fe})_b} \quad (3)$$

where the $(C_i/C_{Fe})_s$ is the ratio of concentration of the ith PTM to the concentration of Fe in the sample, and the $(C_i/C_{Fe})_b$ shows the ratio of concentration of the ith PTM to the concentration of Fe of the considered background reference values.

2.5.3. Contamination Factor (CF)

CF is the ratio of PTM concentration to its reference concentration, which is used to reveal the contamination status of the sediment with the particular PTM [34], and is calculated according to Equation (4).

$$CF = \frac{C_i}{B_i} \quad (4)$$

where C_i is the content of the metal and B_i is the pre-industrial value of the metal.

2.5.4. Pollution Load Index (PLI)

The pollution load index (PLI) describes the cumulative impact of the PTM pollution level. It was proposed by Tomlinson et al. [35], and classified as either pollution or no pollution caused by PTMs. It involves the calculation of CFs of PTMs. The calculation of PLI is given below as;

$$PLI = \left(\prod_{i=1}^{n} CF_{Ei}\right)^{1/n} \tag{5}$$

where CF_{Ei} indicates the mean value of the contamination factor of the ith metal and n is the total number of PTMs [27].

2.5.5. Potential Ecological Risk Index (PERI)

The PERI was initially proposed by Hakanson et al. [36] to demonstrate the ecological risks of PTMs, and has been widely used for riverine sediment samples. In the present study, a modified PERI (mPERI), used earlier by several studies [4,37], was employed, which involved CF values of PTMs in its calculations. The equations for the calculations of mPERI are given below:

$$Er_i = \sum_{i=1}^{n} (Tr_i \times CF_i) \tag{6}$$

$$mPERI = \sum_{i=1}^{n} Er_i \tag{7}$$

where Er_i is the ecological risk index of the ith PTM, which involves the CF of the respective PTM (CF_i), and Tr_i represents the toxicity response coefficient of the respective PTM. The toxic response coefficients used for several PTMs were 10, 30, 5, 2, 5, 40, 5, 5, 7, and 1 for As, Cd, Co, Cr, Cu, Hg, Ni, Pb, Sb, and Zn [38], respectively.

2.5.6. Integrated Toxic Risk Index (ITRI)

The ITRI is a newly developed method that provides an integrated assessment of trace-element toxicity in the environment, considering threshold effect level (TEL) and probable effect level (PEL) values. The ITRI values in sediment samples were computed as

$$ITRI = \sqrt{\frac{\left(\frac{C_i}{TEL_i}\right)^2 + \left(\frac{C_i}{PEL_i}\right)^2}{2}} \tag{8}$$

where C_i symbolizes a particular PTM concentration.

2.5.7. Nemerow Pollution Index (NPI)

NPI is used to measure the overall metal contamination level of multiple PTMs in the sediment [39]. NPI was calculated for PTMs in the sediments, as follows:

$$NPI = \sqrt{\frac{(CF_{mean})^2 + (CF_{max})^2}{2}} \tag{9}$$

where CF_{mean} and CF_{max} are the mean and maximum values of the CF of all the metals studied.

Since the local standardized background values representative of the study area are not yet established, the average shale values from [34] were opted for, to consider as background values of PTMs in the calculations of included pollution assessment methods in this study. However, the interpretation of value ranges and descriptions of pollution levels for the methods used is given in Table S3.

2.5.8. Sediment Quality Guidelines (SQGs)

The SQGs aimed to provide benchmarks for assessing the quality of sediments and identifying potential adverse effects of PTMs on aquatic organisms and ecosystems. This approach has immense significance in regard to evaluating the tolerable concentrations of PTMs in sediments, by comparing them with established quality guidelines [4]. Two sets of sediment quality guidelines (SQGs), namely ERL/ERM (effect range low/effect range median) and TEL/PEL (threshold effect levels/probable effect levels), were employed for the assessment of sediment quality, as presented in Table S3. Contaminant concentrations were sorted into three classes of SQGs. For instance, the PTM values, such as less than ERL and/or TEL, in between ERL-ERM and/or TEL-PEL, and greater than ERL and/or TEL, indicate rare, occasional, and frequent adverse effects, respectively, likely to occur in the aquatic ecosystem.

2.5.9. Modified Hazard Quotient (mHQ)

The mHQ is used to assess the sediment pollution impact from PTM contamination [40]. mHQ measures PTM risk for organisms using thresholds TEL, PEL, and SEL. mHQ can be calculated as follows:

$$mHQ = \sqrt{C_i \left(\frac{1}{TEL_i} + \frac{1}{PEL_i} + \frac{1}{SEL_i} \right)} \quad (10)$$

where C_i is investigated PTM concentration, TEL_i is the threshold effect level, PEL_i is the probable effect level, and SEL_i is the severe effect level, respectively. The values of TEL, PEL and SEL are provided in Table S3.

3. Results and Discussion

3.1. PTM Concentrations in Sediments

Descriptive statistics of PTM concentrations in sediments of the River Ravi, Lahore, are provided in Table 1. The results indicated that mean concentrations (mg kg^{-1}) of PTMs ranked as Al (36503 ± 5433) > Fe (30859 ± 4467) > Mn (297 ± 38.6) > Cr (128 ± 35.6) > Zn (81.3 ± 20.2) > Sr (32.8 ± 6.86) > Pb (26.6 ± 5.58) > Cu (26.5 ± 8.7) > Hg (16.6 ± 4.59) > V (16.4 ± 3.44) > Ni (14.6 ± 3.13) > As (12.8 ± 1.26) > Co (9.42 ± 1.25) > Sb (2.44 ± 1.09) > Cd (2.37 ± 0.41) > Sn (1.81 ± 0.46). The results of the present study showed that most of the PTMs, including Al, As, Co, Cu, Fe, Mn, Ni, Sn, Sr, V, and Zn, have concentrations lower than the background value of shales. However, the mean concentrations of Cd, Cr, Hg, Pb, and Sb were found to be higher than the background value of shales (Table 1). A previous study based on the heavy-metal assessment in the sediments of the River Ravi by [41] found the minimum and maximum concentrations (mg kg^{-1}) of PTMs in the order of Cu (0.00338–0.15979) > Cr (0.0046–0.0574) > Cd (0.00099–0.00317) > Co (0.00222–0.01853); comparative to this, the range of Cu, Cr, Cd, and Co found in the present study was considerably higher. Similarly, the mean values (mg kg^{-1}) of Hg, Pb, Ni, Cr, Cu, and Zn found in the present study were also higher than Hg (1.9), Pb (130.7), Ni (165.3), Cr (16.1), Cu (14.8), and Zn (159.7) in the sediments of the Ravi River reported by a previous study conducted on the riverine ecosystem of Pakistan [3]. The results of a study by Javed [42] revealed lower concentrations of Zn, Pb, and Ni in sediments of River Ravi, as the concentration of the current study showed higher values, which may be due to industrial input or uneven discharge from commercial locations. However, extended exposure to PTMs can pose significant risks to aquatic ecosystems and the local population [43].

Table 1. Statistical summary of PTM concentrations in sediments of River Ravi, Lahore, and their comparison with reference values.

	Potentially Toxic Metals	Al	As	Cd	Co	Cr	Cu	Fe	Hg	Mn	Ni	Pb	Sb	Sn	Sr	V	Zn
									mg kg^{-1}								
Statistics of PTMs	**Minimum**	27,000	10.7	1.85	7.54	76	17.2	25,100	8.61	250	11.5	16.1	0.99	1.08	19.7	11.5	50.9
	Maximum	56,500	15.5	3.37	12.86	230	57.5	47,200	28.75	386	26.4	41.0	6.41	3.29	43.2	27.1	150.4
	Mean	36,500	12.8	2.37	9.42	130	26.5	30,900	16.56	297	14.6	26.6	2.44	1.81	32.8	16.4	81.3
	SD	5430	1.3	0.41	1.22	36	8.7	4470	4.59	39	3.1	5.6	1.09	0.46	6.9	3.4	20.2
	Median	36,300	12.5	2.27	9.23	120	24.0	30,300	16.73	290	14.2	25.9	2.24	1.77	32.7	15.7	78.9
Reference values	**Shales** [a]	80,000	13.0	0.30	19.0	90.0	45.0	47,200	0.40	850	68.0	20.0	1.50	6.18	300	130	95.0
	UCC [b]	81,500	4.8	0.09	17.3	92.0	28.0	39,200	0.05	774	47.0	17.0	0.40	2.10	320	97.0	67.0
	WSA [c]	NA	4.7	1.10	6.90	42.0	14.0	NA	0.10	418	18.0	25.0	0.62	NA	147	60.0	62.0

[a] [34]; [b] [44]; [c] [45]; UCC = upper continental crust; WSA = world soil average.

Furthermore, the concentrations of PTMs were evaluated against established SQGs for sediments, as given in Table 2. The SQGs comparisons were conducted exclusively for As, Cd, Cr, Cu, Hg, Ni, Pb, and Zn, owing to the absence of reference guidelines for other PTMs [46]. Among these PTMs, Cu, Ni, Pb, and Zn concentrations were observed to be below the threshold effect level (TEL), indicating rare adverse effects. However, occasional adverse effects were noted for As, Cd, and Cr. The prevalence of frequent adverse effects attributable to Hg was particularly concerning, with 96% of its value exceeding the probable effects level (PEL).

Table 2. Percent proportions of total collected samples representing the corresponding quality guidelines for selected PTMs.

PTMs	<ERL	ERL-ERM	>ERM	<TEL	TEL-PEL	>PEL
As	0%	100%	0%	0%	100%	0%
Cd	0%	100%	0%	0%	100%	0%
Cr	48%	48%	4%	0%	88%	12%
Cu	92%	8%	0%	16%	84%	0%
Hg	0%	4%	96%	0%	4%	96%
Ni	96%	4%	0%	88%	12%	0%
Pb	100%	0%	0%	76%	24%	0%
Zn	96%	4%	0%	96%	4%	0%

ERL = effect range low, ERM = effect range medium, TEL = threshold effect level, and PEL = probable effect level.

3.2. Pollution Levels, Ecological Risk, and Toxicity Risk of PTMs

3.2.1. Geo-Accumulation Index (I_{geo})

Calculated mean I_{geo} values of PTMs are in the order of Hg> Cd > Sb > Pb > Cr > As > Zn > Fe > Cu > Co > Al > Mn > Sn > Ni > V > Sr (Figure 2). The geo-accumulation indices were calculated based on the shale values reported for the upper continental crust. According to Müller's classification of I_{geo} values, no accumulation class was observed for Al, As, Cd, Co, Cr, Cu, Fe, Hg, Mn, Ni, Pb, Sb, Sn, Sr, V, and Zn for having $I_{geo} < 0$. However, moderate-to-heavy accumulation ($2 < I_{geo} \leq 3$) existed for Cd, having an I_{geo} value of 2.37, and heavy-to-extreme accumulation ($4 < I_{geo} \leq 5$) was estimated for Hg with an I_{geo} value of 4.54.

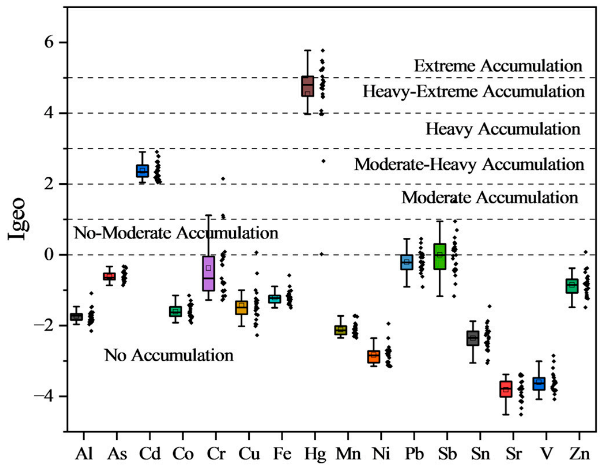

Figure 2. The box plot shows the mean, median, and range of the geo-accumulation index (I_{geo}) for various PTMs.

3.2.2. Enrichment Factor (EF)

According to the EF calculations (Figure 3a), minimum enrichment was found for Al, As, Co, Cu, Fe, Mn, Ni, Sn, Sr, V, and Zn, having EF values in the range of 0 < EF ≤ 2. However, moderate enrichment (2 < EF ≤ 5) was observed for Cr, Pb, and Sb, having EF values of 2.27, 2.04, and 2.52, respectively; significant enrichment (5 < EF ≤ 20) was the case with Cd, with the EF value of 12.10, and extreme enrichment (EF > 40) was posed for Hg, having an EF value of 63.20.

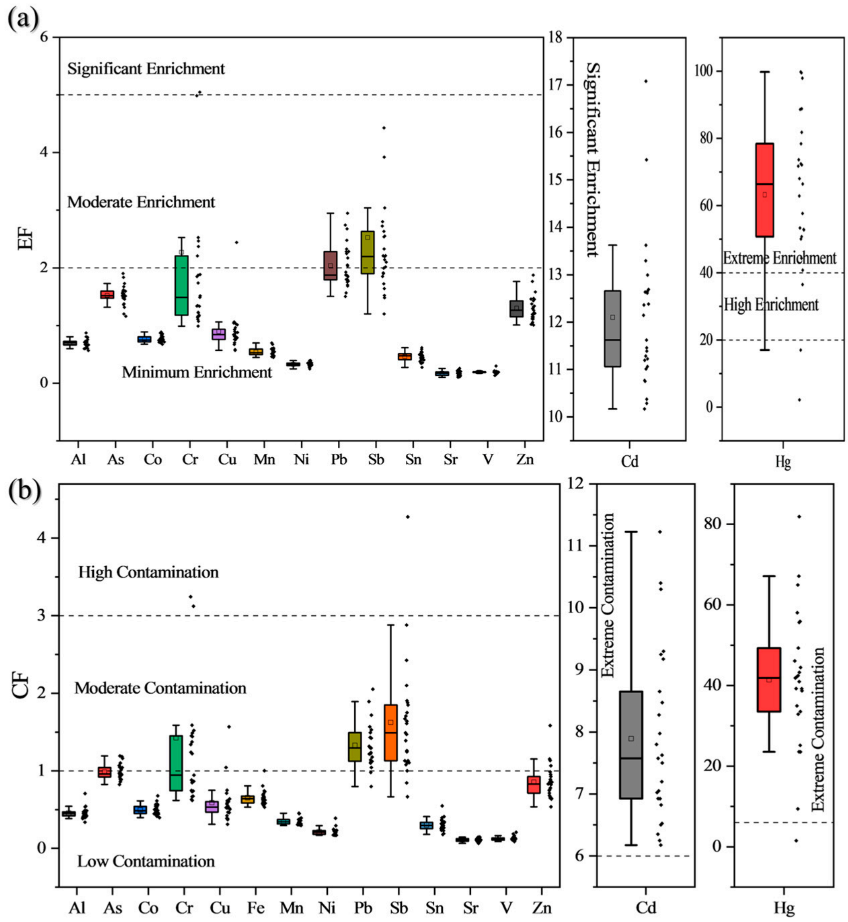

Figure 3. The box plots show the mean, median, and range of values for (**a**) enrichment factors (EFs) and (**b**) contamination factors (CFs) of various PTMs.

3.2.3. Contamination Factor (CF)

As a result of CF calculations, Hg (41.4) and Cd (7.89) showed extreme contamination (CF > 6), while moderate contamination (1 < CF ≤ 3) was estimated for Sb (1.62) and Pb (1.33), and no contamination was found for the rest of the PTMs, including Al, As, Co, Cr, Cu, Fe, Mn, Ni, Sn, Sr, V and Zn (Figure 3b).

3.2.4. Modified Hazard Quotient (mHQ)

The results for mHQ showed that Hg has the extreme (3.5 < mHQ) level of contamination hazard, based on the value 12.2. However, significant (2 < mHQ < 2.5) contamination existed for Cd (2.06). In addition to this, moderate (1.5 < mHQ < 2) hazard was the case for Cr and As, having mHQ values of 1.98 and 1.57. However, Cu, Ni, Pb, and Zn also showed low contamination (1 < mHQ < 1.5) (Figure S1).

3.2.5. Nemerow Pollution Index (NPI)

The NPI results revealed that in river sediments, no pollution was observed for Al, Co, Fe, Mn, Ni, Sn, Sr, and V (NPI < 1). However, slight pollution (1 < NPI < 2.5) was observed due to As, Cu, Pb, and Zn, while moderate pollution (2.5 < NPI < 7) was reported for Cr (4.81) and Sb (3.23). Heavy pollution (NPI > 7) in the sediments was revealed, due to elevated levels of Hg (64.9) and Cd (9.70) (Figure 4). These results underscore the urgent need for swift and comprehensive environmental action.

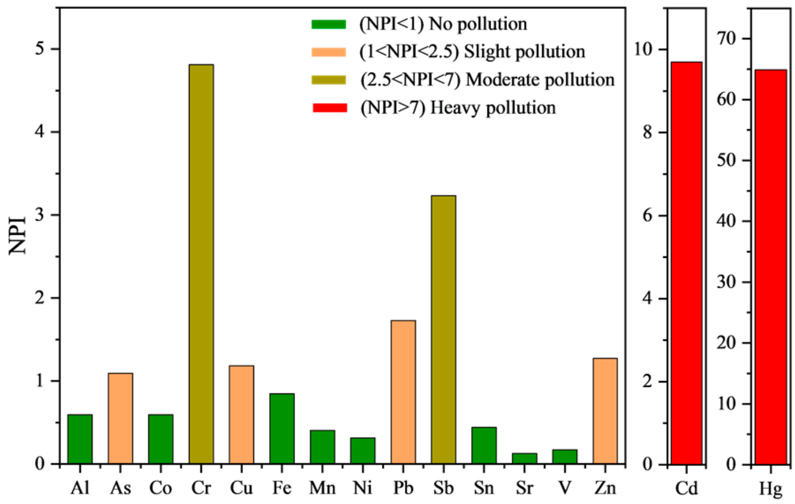

Figure 4. Bar graph illustrations reveal the alarming values of the Nemerow pollution index (NPI), especially for chromium (Cr), antimony (Sb), cadmium (Cd) and mercury (Hg) in the sediments of River Ravi, Punjab, Pakistan.

3.2.6. Ecological and Toxicity Risk Assessment

Calculated Er_i values showed extreme ecological risk ($Er_i \geq 320$) for Hg (1656), high ecological risk ($160 \leq Er_i < 320$) for Cd (237), and low ecological risk for the rest of the PTMs (Figure 5a). Compared to this, a study stated Pb, with Er_i value of 131, as the main factor for highest ecological risk followed by Hg, with Er_i value of 95, in the River Ravi sediments [3]. The calculated values for ITRI showed extreme toxicity risk (TRI > 20) for Hg, with a value of 91.6. However, As, Cd, Cr, Cu, Ni, Pb, and Zn posed no toxicity risk (TRI ≤ 5) (Figure 5b). Evaluation of PTMs based on the combined risk assessment indices by PLI, NPI, and TRI are provided in Figure S2, and the pie chart of PERI is given in Figure S3. The illustrations showed that the PTM pollution in the Ravi sediments posed potential ecological risks, predominantly due to Hg contributing 85.8%, followed by Cd with 12.3%, to overall PERI.

Sediments are a major source of multiple pollutants, including pesticides, toxic metals, and organic matter, and, therefore, play a significant role in the fate and transport of contaminants in aquatic environments under favorable conditions. Additionally, the enrichment of minerals from the underlying geological substratum also alters the concentrations of metals in the river sediments [41]. Therefore, this study meticulously assessed the pollution status, ecotoxicological risks, and identification of their potential sources from the sediments of River Ravi. Overall pollution indices, including I_{geo}, EF, and CF, consistently indicated moderate Cd pollution and extreme Hg pollution. Likewise, Eri and ITRI also justified the fact that ecological and toxicity risks were extreme, due to elevated pollution levels of Cd and Hg. These findings showed significant input of anthropogenic activities, particularly for Cd and Hg, which align with previous pollution assessments of PTMs in sediments of River Ravi [3]. However, the pollution levels of other PTMs in sediments were relatively less, suggesting minimal-to-no human-induced impacts [47]. Urban areas are known to contain elevated concentrations of PTMs, which result primarily from anthropogenic activities [27,48]. Lahore city, in particular, has gained much attention as one of the most polluted cities in the world, with high levels of industrial activities. The potential contamination load of wastewater generated by municipal and industrial sources contributes significantly to PTM pollution in the River Ravi. Cd and Hg can potentially be sourced from industries such as plastic production, metallic ware, batteries, and paints and pigment manufacturing, which spread in small clusters throughout the urban area of Lahore.

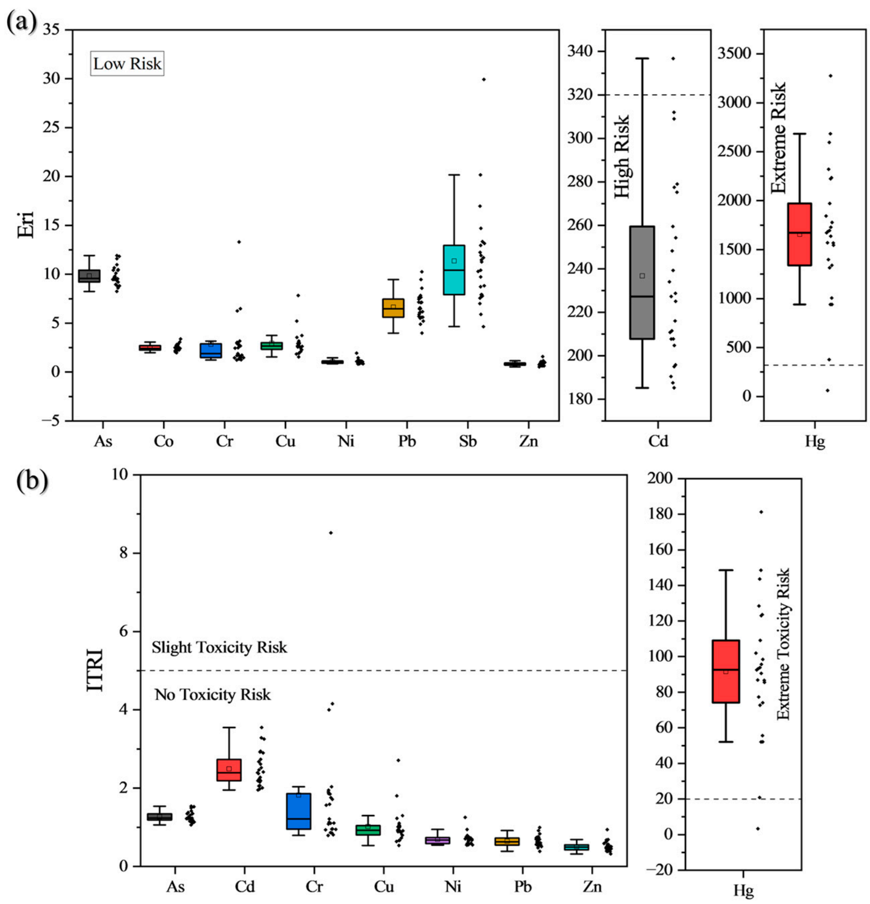

Figure 5. The box plots show the mean, median, and range of values for (**a**) the Ecological Risk Index (Eri) and (**b**) the Integrated Toxicity Risk Index (ITRI) of PTMs.

3.3. XRD and SEM-EDS Results

The mineral composition of sediments has an immense influence on the mobility of PTMs in the aquatic ecosystem. Different minerals have an affinity to adsorb PTMs, depending upon the exchange-site availability offered by the minerals, especially clays [49]. Importantly, the exchange sites for minerals have become more vulnerable, through increased hydration in the riverine environment [50]. The XRD results showed quartz dominance, with contributions from kaolinite, feldspar, and calcite minerals (Figure 6). Dominance of quartz in sediments is general, which may hinder the immobilization and hence increase the bioavailability of the PTMs, especially Cd and Zn in the riverine ecosystem [51]. However, clay minerals comprising iron–aluminum silicates, i.e., kaolinite and feldspar, have high surface-area and charge sites, resulting in high adsorption of PTMs like Cd, Cr, Cu, Pb, and Zn [52]. Though PTMs containing minerals have not been dominant in riverine sediments, the capability of these minerals to hold PTMs with back-and-forth immobilization and mineralization transformations could be crucial for their bioavailability and toxicity for aquatic biota in the riverine ecosystem.

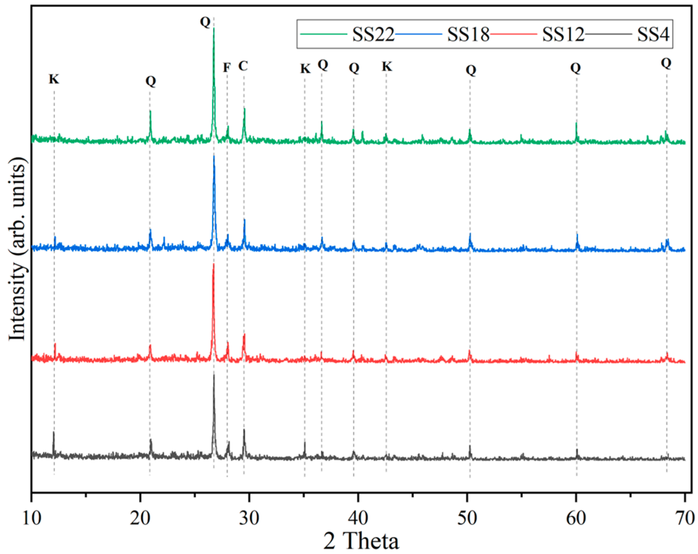

Figure 6. X-ray diffraction analysis (XRD) spectra for representative sediment samples (SS4, SS12, SS18, SS22) showing corresponding peaks for a range of minerals, including Q = Quartz, K = Kaolinite, F = Feldspar, and C = Calcite.

The SEM images were taken at various magnifications to study morphology and anthropogenic influence on the sediments, and results are depicted in Figures 7 and S4. The observations under SEM lenses depicted those sediments carrying several shapes of particles, with varying particle sizes. However, the EDS spectra showed that relatively smaller particles were carrying silicon and aluminum elements, indicating that the association with aluminosilicates comes from the weathering process of minerals and rocks. Overall, sediments predominantly contain elements like Si, Mg, Al, Ca, Ti, and V, which can be associated with those naturally concentrated in the Earth's crust [53,54], while the anthropogenic particles can be distinguished and spotted in Figures 7 and S4, predominantly carrying PTMs like Cu, Fe, Pb, and Zn. Clearly, anthropogenic input was evident in distinguished particles in sediments. Local characteristics of the sampling area were observed for anthropogenic influence on the River Ravi. Predominantly, roadside activities, plastic factories, agricultural farmland areas, boating, picnic points, and ceramic processing units were observed widely, and were perhaps the source of anthropogenic influence. However, these anthropogenic activities can be associated with potential sources of PTMs in the sediments [55,56].

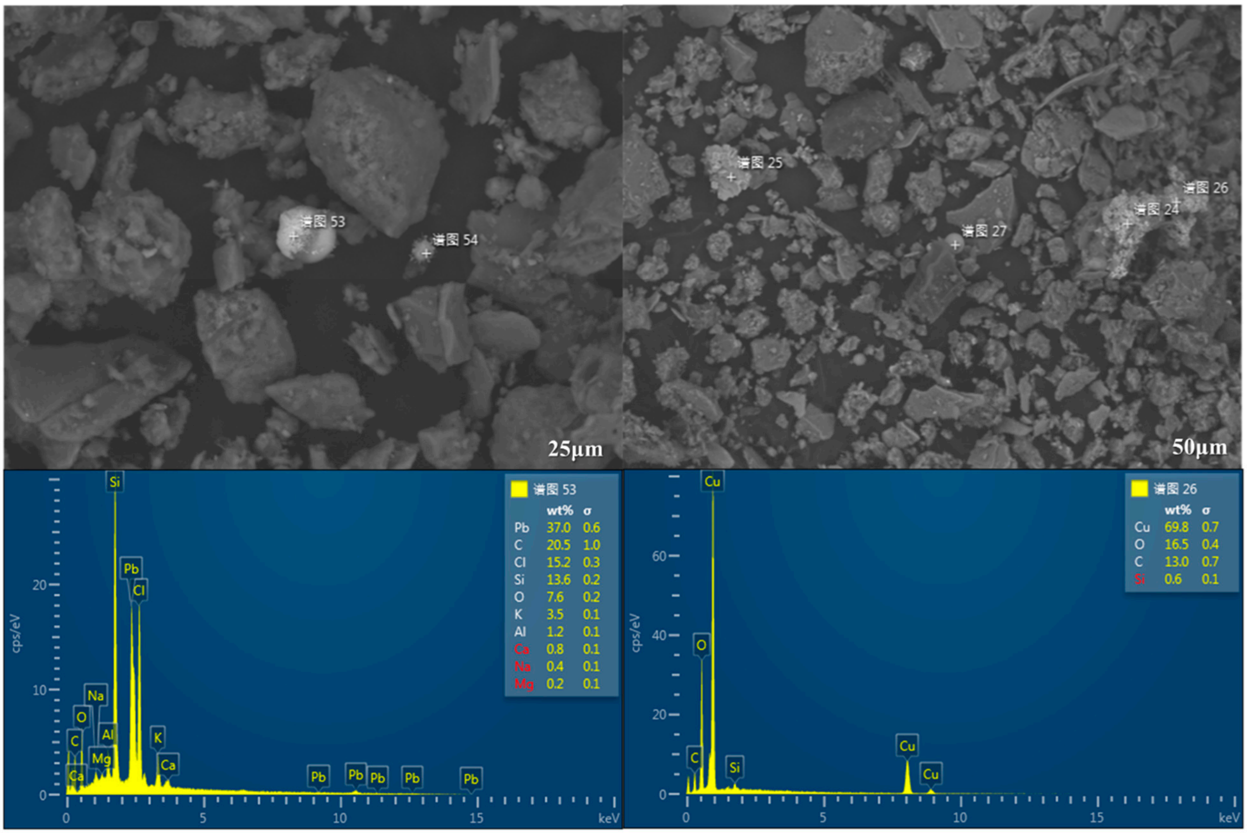

Figure 7. Scanning electron microscopy (SEM) images show the anthropogenic influence in the riverine sediments, by showing distinguished particles. EDS spectra indicated the quantitative proportion of various PTMs from corresponding particles.

3.4. Association Among PTMs

PCC was performed to correlate the relationship among PTMs in the river sediments. The PCC among the concentrations of PTMs (Al, As, Cd, Co, Cr, Cu, Fe, Hg, Mn, Ni, Pb, Sb, Sn, Sr, V, and Zn) in the sediments is depicted in Figure 8a. The corrplot illustrates that V and Ni (r = 0.91), Sb and Cr (r = 0.90), Sb and Fe (r = 0.88), Ni and Fe (0.88), Sn and Ni (r = 0.85), Fe and Co (r = 0.84), and Fe and Al (r = 0.78) were correlated positively with each other, having strong statistical association ($p < 0.05$). However, a negative correlation was observed between Pb and Cr (r = −0.43) ($p < 0.05$). Cr has also shown a weak negative relationship with Al, As, Cd, and Co. The substantial presence of positive correlations among PTMs in the river sediments suggests the influence of various anthropogenic sources. These may include improper waste disposal, discharge from industrial activities, agricultural practices, inadequate sanitation, and organic decomposition within the study area [57]. CA was used to identify similarities or differences among observations, represented through a hierarchical clustering diagram. The results demonstrated the grouping of observations into two prominent clusters, with sampling site S-9 (boating point) forming a separate group, due to its significantly high pollution levels caused by PTMs (Figure 8b). The other leading groups divided into three sub-groups, reflecting clustering based on land use (see Table S1). For instance, S-2, S-14, and S-21 are grouped together, as they share the same land-use type, i.e., agricultural land. Similarly, areas with higher anthropogenic influences, such as traffic and industrial disturbances, are clustered in the group comprising S-5 to S-10. Sites impacted by residential and agricultural activities, including S-15, S-20, and S-25, form another distinct cluster. Lastly, sites influenced by municipal waste (S-19) and plastic manufacturing (S-11) are grouped together, suggesting similar sources of pollution. The PCA can usually demonstrate the presence of positive and negative relationships between variables in complex datasets, facilitating the identification of the similarities in behaviors among different PTMs and their sources of contamination [58]. The results of PCA, including three components having eigenvalues higher than 1, percent variance higher than 10%, and cumulative variance up to 75.6% with eigenvector coefficients, are summarized in Table S4 and are well-depicted in Figure 8c. Apparently, the result of the PCs corresponds well with the PCC illustrations. The first principal component (PC1), with a variance of 49.4%, was highly correlated with Fe, Ni, Co, V, Sn, and Zn, represented by high eigenvector coefficients (0.323, 0.320, 0.311, 0.315, 0.295, and 0.293, respectively), which showed the balanced contribution of these PTMs to PC1. The second principal component (PC2) accounted for a variance of 14.6%, and was largely contributed by Mn, Sb, Cr, and Sr, having eigenvector coefficients of 0.427, 0.397, 0.39, and 0.341, respectively. Interestingly, the third principal component (PC3) has the highest coefficients of Sb (0.569) and Cr (0.524), representing the strongest influence of these two PTMs, corresponding to about 11.6% of the total variance (Table S4). The prominent PTMs in all three principal components contributed almost equally to their respective components, which suggests that multiple sources contributed to the PTM concentrations in the sediments, but none was overwhelmingly dominant.

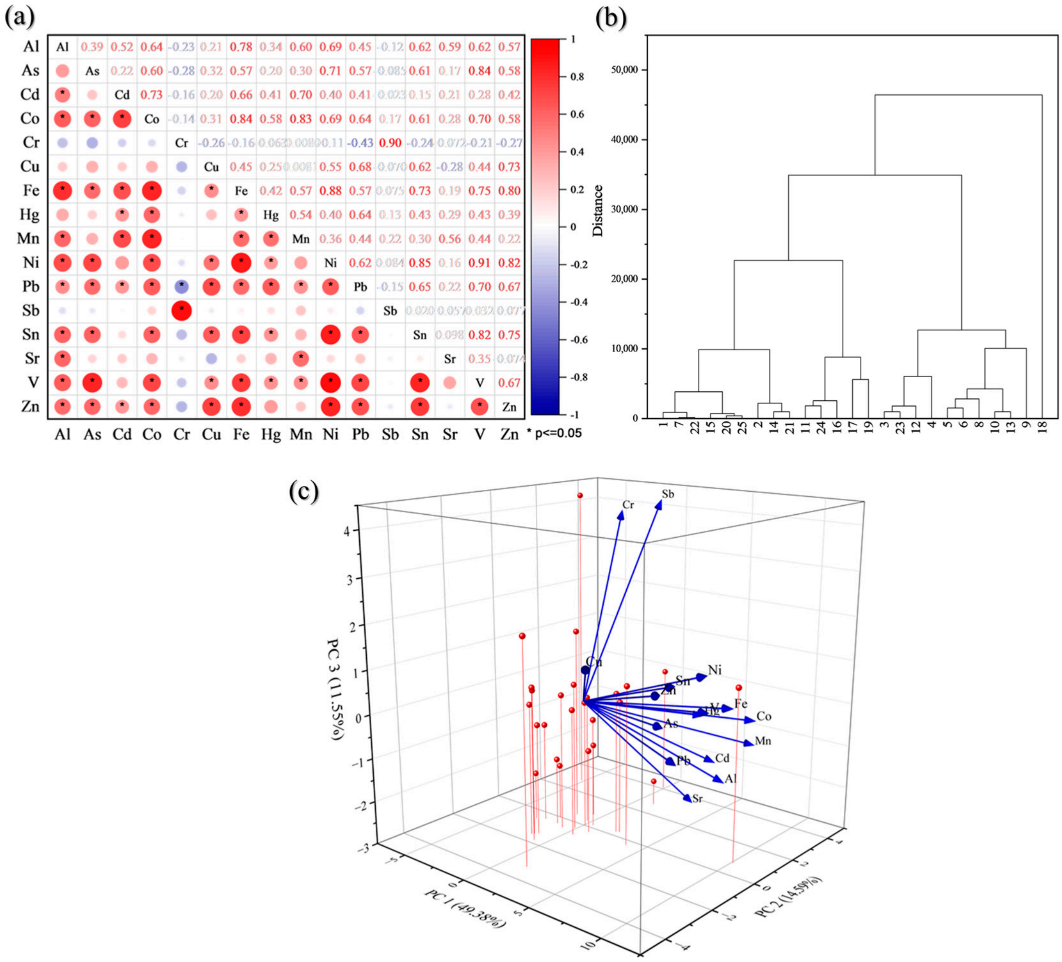

Figure 8. Results of multivariate statistical analysis illustrating the (**a**) corrplot of Pearson's correlation coefficient (PCC) at $p < 0.05$; (**b**) cluster analysis (CA) dendrogram; and (**c**) principal component analysis (PCA) using the rotation method (varimax with Kaiser normalization).

3.5. Anthropogenic Sources and Impact of Land Use on PTM Pollution

The main sources of pollution in the River Ravi comprise urban, agricultural, and industrial wastewater. These pollutants are derived from various origins, including electroplating workshops, steel factories, paper and pulp industries, medicine, and scientific laboratories. Moreover, surface runoff and municipal sewage contribute significantly to river contamination [41]. Atmospheric deposition of PTMs in aquatic environments is also a major source of pollution, resulting from the sinking of PTMs in the riverbed sediments [21]. The results of PTM concentration at each sampling site of the study area provide an overall outlook for comprehending the pollution source pathways and the interlinked land-use impact, as described in Table S1. The elevated concentrations of most PTMs, including Al, Fe, Co, Hg, Ni, Pb, V, and Zn, were characterized at sampling site S-18 (Mohlanwal). Multivariate statistical analyses also corroborated the association among these PTMs, which were found at close distances in CA and relatively higher factor loadings in PC1 (Table S4). Locals have reported that the boating points at both riverbanks are commonly used for transporting agricultural products, local vehicles, and other goods through small boats. Contrastingly, sampling site S-9 (industrial area) showed that not many of the concentrations of PTMs were found at this site, except, interestingly, that of Cr and Sb. Results of multivariate analysis (Figure 8) were also concomitant with this finding and revealed that Cr and Sb are the major contributing PTMs in PC3, with eigenvector coefficients higher than 0.5. However, these findings were also in line with the results of I_{geo} and EF, which showed the accumulation and enrichment of Hg and Cd, also indicating the higher concentrations of Cr and Sb, which might be due to the natural sources, including the weathering of underlying substratum and the deposition from the flow of water and mineral containing sediments from upstream rivers [19]. Since PC1 and PC2 showed relatively lower eigenvector coefficients for Fe, Ni, Co, Al, Mn, Sb, and Sr, this may indicate the natural origins of parent-rock materials and mineralized ore deposits, attributed to processes of weathering and erosion [2].

4. Conclusions

The present study was designed to assess the pollution, ecotoxicological risks, and source identification of PTMs in surface sediments of the River Ravi, using multivariate analytical and statistical tools. Several pollution assessment indices were utilized to estimate the pollution level, and multivariate statistical methods for source identification were supported by XRD and SEM-EDS analyses of PTMs from sediment collected from the urban riverine stream of Lahore. The sediments of River Ravi were heavily contaminated with mercury (Hg) and cadmium (Cd), with concentrations exceeding background reference values. The findings of several pollution indices, including I_{geo}, EF, and PLI, also showed severe Hg and moderate Cd pollution, indicating the presence of human-induced impact on these PTMs. The elevated pollution load of PTMs and ecological and toxicological risk were also high, predominantly due to Hg and Cd pollution, which pose immense threats to aquatic life. The mineralogical study of the sediments provided limited insights into the immobilization of PTMs in the riverine ecosystem, while SEM-EDS results illustrated a small extent of anthropogenic influence, as sediment samples contained anthropogenic particles. However, multivariate statistical analyses showed associations among PTMs and sampling locations, which revealed that agricultural practices and industrial activities could be potential sources of PTMs in the sediments of the River Ravi. To combat PTMs contamination, urgent action is needed, including stricter pollution controls, enhanced industrial wastewater treatment, fast development of remediation strategies, and robust regulations on discharging pollutants like PTMs into the terrestrial environment. Furthermore, public awareness and targeted health measures, especially for vulnerable groups like children, are essential to mitigate risks and protect the environment and public health.

Supplementary Materials: The following supporting information can be downloaded at: https://www.mdpi.com/article/10.3390/land14010032/s1. Refs. [59,60] are cited in the file, too.

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