

Original Article

Preliminary Assessment of Several Heavy Metal Ions (Fe, Cu, Ni, Zn, Cr, Pb, and Cd) in Water, Sediment, *Ceratophyllum demersum*, and *Potamogeton pectinatus* Plants from Marsh Al-Hawizeh, Iraq

Al-thahaibawi B.m.h.

Directorate Environment of Maysan, Ministry of Health and Environment Iraqi, Maysan, Iraq

ABSTRACT

Seasonal samples of water, sediments, and two submerged plants, *Ceratophyllum demersum* and *Potamogeton pectinatus*, were collected from four sites within Al-Hawizeh marsh between December 2017 and November 2018 to assess several heavy metal concentrations (Fe, Cu, Ni, Cd, Pb, Cr, and Zn) and analyzed by using Flame Atomic Absorption Spectrophotometer. All concentrations were compared to permissible limits for aquatic life. The findings have clarified levels of metals so as the following: Sediments > plants > water. Concentrations of heavy metals in sediment decreased within the sequence Fe > Ni > Cd > Cu > Cr > Zn > Pb (mg/kg), plants as Fe > Zn > Cd > Cr > Ni > Pb > Cu (mg/kg dry weight), in water as Fe > Ni > Zn > Pb > Cr > Cd (mg/L). The concentrations of all metals in water didn't exceed the permissible limit, thus Fe, Ni, and Pb showed a greater concentration in water samples than other metals. In sediments Fe, Ni, and Cd have exceeded the permissible limits except Cd wasn't detected (ND) during the winter, whereas, Zn and Pb didn't exceed the recommended limits. In-plant samples as *C. demersum* Fe, Cd, Zn, and Cr have exceeded the allowable limits, while Cu and Pb didn't exceed the permissible limits, also in *P. pectinatus* Fe, Cd, and Zn have exceeded the permissible limits, whereas, Ni, Cu, Cr, and Pb didn't exceed the permissible limits altogether seasons during this study. The correlation of Pearson was administered to detect the interrelationship between metal concentrations. A significant positive correlation ($p \leq 0.05$) was found in sediment rather than plants and water samples.

Keywords: bioaccumulation, bioconcentration, macrophytes, heavy metals, marsh

INTRODUCTION

At certain amounts, heavy metals are essential or advantageous in nature, but also, they pose a threat to human health if their concentrations exceed the permissible limit [1]. Compositions of heavy metals vary across different localities, leading to spatial differences of surrounding concentrations [2]. As a result of rapid growth and anthropogenic activities, like poorly treated industrial, domestic sewage, increased agricultural runoff, construction, and mining heavy metals are released into lakes and other water bodies [3]. Usually, urban runoff includes a spread of contaminants that might reduce the standard of water, also physical and chemical

associated changes could restructure biotic ecosystems and reduce the range and productivity of invertebrates and fish within the aquatic ecosystem [4]. Furthermore, metals enter these aquatic environments mainly through natural inputs like weathering, erosion of rocks, and discharge into water [5] and typically end up in the sediments [6]. Also, urban stormwater carries vast quantities of metals into water sources [7].

Due to their bioaccumulation, environmental threats, toxicity, persistence, and potential to be introduced into food chains, heavy metals in water are a worldwide concern [8], also they're considered to be significant contaminants [9]. However, some heavy metals like Cu, Fe, Mn, Ni, and Zn are

Corresponding author: Al-thahaibawi B.m.h., E-mail: basim.althahaibawi74@gmail.com

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Table 1 Geographical positioning system (GPS) and some characteristics of each site in the study area.

Site	Location	Latitude (North)	Longitude (East)	Vegetation and activities
Site 1	Umm Al-Ward	47° 32' 9".15	31° 33' 45".50	Low vegetation, less human activities, and presence of fisherman
Site 2	Um Al-Nia-aj	47° 38' 22".50	31° 35' 45".15	Med vegetation, (Open area) and high presence fisherman
Site 3	Al-Souda north	47° 39' 50".38	31° 40' 22".26	Low vegetation, less human activities, and presence of fisherman
Site 4	Al-Adaim	47° 45' 35".40	31° 41' 22".36	Low vegetation, less human activities, and Nonpresence fisherman

important as micronutrients for life processes in very limited quantities in animals and plants, additionally to other metals like Cd, Cr, Hg, and Pb have unknown physiological activity and are extremely toxic even at trace levels [10]. Nevertheless, metals can undergo regular modifications during their transport within the riverine environment due to dissolution, precipitation, and sorption phenomena [11], thus affecting their performance and bioavailability [12].

Heavy metals accumulate within the sediments through complex physical and chemical adsorption processes relying on the character of the sediment matrix and therefore the properties of the adsorbed compounds [13]. Moreover, the build-up and releasing of metals in sediments is regulated by their geochemistry, especially the shape and quantities of organic matter, grain size, and capacity for cation exchange [14]. Once they're released in marine environments, heavy metals are normally bound to particulate, which subsequently settle down and dissolved into sediments. Additionally, surface sediments in marine habitats are the foremost source of metals and other contaminants [15].

The load of heavy metals causes an imbalance in aquatic environments and thus the biota emerging under such habitats can accumulate high amounts of heavy metals (Cu, Zn, Cd, Cr, Ni, etc.) which are successively assimilated and transferred by the magnification mechanism into food chains [16]. Knowing how metals are stored, dispersed, and metabolized in aquatic macrophytes has great ecological, scientific and practical significance.

The worth from an environmental point of view is expressed within the incontrovertible fact that aquatic macrophytes could also be used as biological pollution indicators [17]. Hence, the main objective of this work is to determine the concentrations of heavy metals in water, sediment, and in two of the submerged plants, *Ceratophyllum demersum* and *Potamogeton pectinatus* of the Al-Hawizeh marsh, because it may be a lively place for several aquatic species like fish, birds, mammals and a source of food for the animals living

during this marsh.

MATERIALS AND METHODS

Description of the research area

Al-Hawizeh marsh lies east of the Tigris River, straddling the Iran-Iraq border, and is located about 70 km from Al-Ammara town. It ranges between (31° 00–31° 45' N, 47° 25'–47° 50' E). Approximately 80 km long, from the Iraqi-Iranian side east of the Tigris River, and 30 km wide [18]. It covers a neighborhood up to 2,400 km², extends then via flood season to cross 3,500 km², and through droughts shrinks to 650 km². The marsh reservoir exceeds approximately 7,000 billion m³ [19]. Al-Hawizeh marsh feeds two large Tigris River distributaries named Al-Musharah and Al-Kahla rivers. Also, water rainfall and water from rivers from Iran, just like the rivers Al-Karkha, Tayib, and Duwayaraj, which discharges into Al-Sanna'f marsh, which empties directly into Al-Hawizeh marsh. During the study period, four sites were selected within the Al-Hawizeh marsh using Geographical Positioning System (GPS). Site 1 was located on the southern side of the Al-Hawizeh marsh near Abu Khasaf village. Site 2 was an open water area located within the center of a marsh. Site 3 was in south site 2 near to Al-Musharah village. Finally, site 4 was located north of the encompassing bordered Iranian marsh (Table 1).

Collection the samples

Sampling sites for water, sediments, and plant species were systematically selected and seasonally collected from four sites within Al-Hawizeh marsh from December 2017 to November 2018 (Fig. 1), for analysis of heavy metal such as iron (Fe), copper (Cu), cadmium (Cd), chromium (Cr), lead (Pb), zinc (Zn), and nickel (Ni).

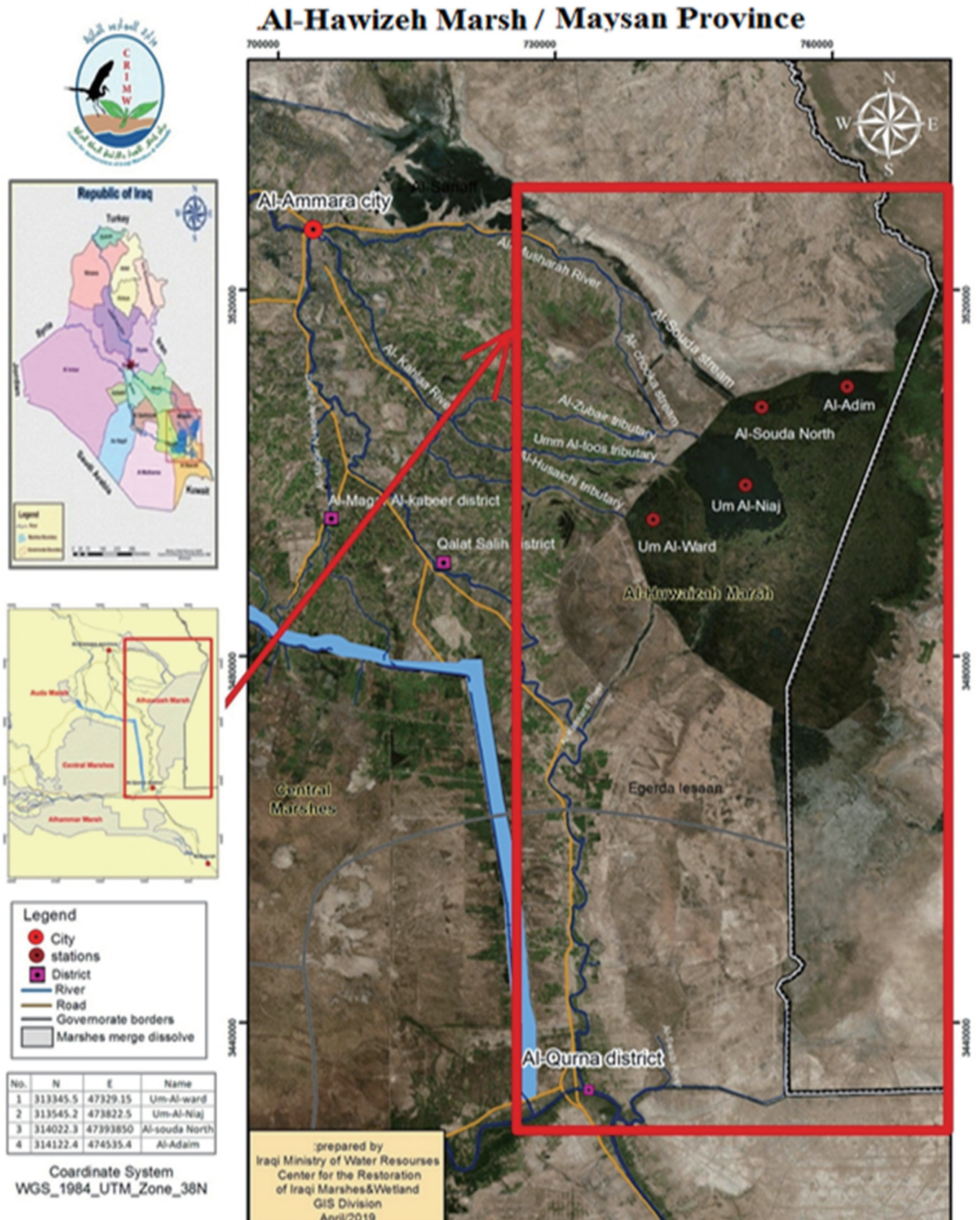


Fig. 1 Study area (Al-Hawizeh marsh) map and sampling sites [20].

ANALYSIS OF SAMPLES

Water samples

Water samples were collected at 20 cm below water surface level and pooled into 1 L of acid-washed polyethylene bottles and delivered to the laboratory, all water samples were filtered directly via millipore filter papers (0.45 μm). These filter papers were washed with hydrochloric acid (5%), deionized water followed and dried at 60°C for 24 hr, filtered water was transferred to a beaker, and acidified with 5 mL of HNO_3 . The beaker was sealed with a ribbed watch bottle; the solution was heated at 95°C on a hot plate until the quantity decreased to around 20 mL. The sample was allowed to cool down and transferred to a volumetric flask of fifty mL. The volume was completed with de-ionized water and allowed to settle for the removal of insoluble materials overnight. Solution has been stored in sterile polyethylene flasks.

Sediments samples

The sediment samples were taken from the study sites using a handheld collector within the surface layer 0–30 cm depth of the bottom sediments. Packed and sealed in pre-washed polyethylene containers, and transported for pretreatment and examination to the laboratory. Samples were air-dried at a temperature within the laboratory, pounded into porcelain mortar and pestle to break down to the aggregates, and digested with nitrohydrochloric acid ($\text{HCl}:\text{HNO}_3$ in 3:1). The solution was evaporated to 2 mL, filtered into a volumetric flask of 20 mL and the final volume was made up to double distilled water markings.

Plant samples

Tested plants are thoroughly washed and rinsed with deionized water using running water [21]. For twenty-four hours, the plant samples were oven-dried at 100°C and mixed with an electrical blender to fineness, then sieved for quick digestion through a 2 mm mesh sieve. 5 mL of 4:1 concentration mixture $\text{HNO}_3:\text{HClO}_4$ has been applied to 1 g of weighted plant with an analytical weighing machine. It had been heated to dryness in 1 hour at a temperature of 105°C [22]. Then allowed left to cool and made with 1 M HNO_3 , up to the 50 mL volumetric flask level. Jena 300 Investigator atomic absorption spectrophotometer (Analytic Jena, Jena, Germany) was used to analyze heavy metals at the Central Environmental Laboratory, Ministry of Health and Environment Iraqi according to APHA (2012) [23].

Statistical Analysis

Statistical analysis was performed by SPSS version 16.0 to calculate mean $\pm$ standard deviation (Mean $\pm$ SD) and Pearson's correlation (r) value to point out the degree of metal association in water, sediment, and plants. The ANOVA test (under the significance level $p \leq 0.05$) was employed to understand the seasonal variation in heavy metal concentrations.

RESULTS

Heavy metals of water

Results of the study showed that the concentrations of heavy metals (Fe, Cu, Ni, Zn, Cr, Pb, and Cd) were varied between samples of studied sites, these heavy metals examined didn't exceed permissible water limits set by [24–26] apart from Pb which was above the permissible limit at both 2 and 3 sites in summer and site 4 within the autumn season. The concentration of metals in water ranked as follows: $\text{Fe} > \text{Ni} > \text{Pb} > \text{Zn} > \text{Cd} > \text{Cu} > \text{Cr}$. Moreover, Fe, Ni, and Pb showed a greater concentration of metals in water samples than other metals (Table 2).

The content of Fe in water ranged from 0.02 mg/L at both 1 and 3 sites during the winter and autumn to 0.12 mg/L at both 1 and 4 sites during the spring. Temporal differences showed the very best 0.091 ± 0.0373 mg/L during the spring, the lowest value 0.028 ± 0.0096 mg/L during the winter. Spatial differences identified the maximum value 0.0675 ± 0.036 mg/L at site 4 and therefore the minimum value 0.0337 ± 0.009 mg/L at site 2 (Table 2). Furthermore, the findings observed significant differences ($p \leq 0.05$) were in Fe values between the study sites and seasons. The concentration of Ni ranged from 0.0018 mg/L at site 2 during the autumn to 0.053 mg/L at site 4 during the summer. The seasonal variations recorded the highest value 0.0213 ± 0.0224 mg/L in the summer, the lowest value 0.0027 ± 0.0009 mg/L in the winter. While the spatial differences detected the maximum value 0.028 ± 0.0201 mg/L at site 4 and the minimum value 0.003 ± 0.0012 mg/L at site 3 (Table 2). However, clear significant differences ($p \leq 0.05$) in Ni values were observed between site 4 during the summer season relative to the opposite sites and seasons.

In the case of Cu, the concentrations ranged from 0.001 mg/L at both 1 and 2 sites in winter and autumn to 0.015 mg/L at site 3 in summer. During the summer, the temporal variations showed the highest value 0.009 ± 0.0049 mg/L, the lowest value 0.002 ± 0.0014 mg/L during the winter. The spatial differences identified the maximum

Table 2 Mean and standard deviation of heavy metals concentration (mg/L) in water.

Heavy Metal	Season	Station 1	Station 2	Station 3	Station 4	Mean $\pm$ SD
Iron (Fe)	Winter	0.02	0.021	0.033	0.04	0.028 $\pm$ 0.0096
	Spring	0.12	0.042	0.081	0.12	0.091 $\pm$ 0.0373
	Summer	0.05	0.032	0.024	0.05	0.039 $\pm$ 0.013
	Autumn	0.05	0.04	0.02	0.06	0.043 $\pm$ 0.0171
	Mean $\pm$ SD	0.06 $\pm$ 0.042	0.0337 $\pm$ 0.009	0.0397 $\pm$ 0.028	0.0675 $\pm$ 0.036	
Nickel (Ni)	Winter	0.002	0.002	0.003	0.004	0.0027 $\pm$ 0.0009
	Spring	0.004	0.003	0.005	0.03	0.0105 $\pm$ 0.013
	Summer	0.007	0.021	0.004	0.053	0.0213 $\pm$ 0.0224
	Autumn	0.002	0.0018	0.0021	0.025	0.0077 $\pm$ 0.0115
	Mean $\pm$ SD	0.004 $\pm$ 0.0023	0.007 $\pm$ 0.0093	0.003 $\pm$ 0.0012	0.028 $\pm$ 0.0201	
Copper (Cu)	Winter	0.001	0.001	0.002	0.004	0.002 $\pm$ 0.0014
	Spring	0.007	0.005	0.003	0.005	0.005 $\pm$ 0.0016
	Summer	0.011	0.006	0.015	0.004	0.009 $\pm$ 0.0049
	Autumn	0.001	0.001	0.012	0.003	0.0042 $\pm$ 0.0052
	Mean $\pm$ SD	0.005 $\pm$ 0.005	0.003 $\pm$ 0.003	0.008 $\pm$ 0.006	0.004 $\pm$ 0.001	
Chromium (Cr)	Winter	0.0002	0.0001	0.0003	0.0002	0.0002 $\pm$ 0.0005
	Spring	0.0002	0.0011	0.0016	0.0023	0.0013 $\pm$ 0.0008
	Summer	0.0008	0.0011	0.0023	0.0062	0.0026 $\pm$ 0.0025
	Autumn	0.0002	0.0005	0.0009	0.0005	0.0005 $\pm$ 0.0003
	Mean $\pm$ SD	0.0004 $\pm$ 0.0003	0.0007 $\pm$ 0.0005	0.0013 $\pm$ 0.0013	0.0023 $\pm$ 0.0023	
Cadmium (Cd)	Winter	0.0001	0.0002	0.0003	0.0003	0.00023 $\pm$ 0.0001
	Spring	0.0002	0.0013	0.002	0.0022	0.00142 $\pm$ 0.0002
	Summer	0.0012	0.0006	0.0013	0.0006	0.00093 $\pm$ 0.0014
	Autumn	0.0005	0.0017	0.0024	0.0016	0.0013 $\pm$ 0.0007
	Mean $\pm$ SD	0.0005 $\pm$ 0.005	0.0009 $\pm$ 0.0006	0.0015 $\pm$ 0.0009	0.0011 $\pm$ 0.0008	
Lead (Pb)	Winter	0.001	0.001	0.001	0.002	0.00125 $\pm$ 0.0005
	Spring	0.002	0.001	0.001	0.003	0.0017 $\pm$ 0.0009
	Summer	0.001	0.015	0.02	0.008	0.011 $\pm$ 0.0082
	Autumn	0.001	0.002	0.002	0.012	0.0043 $\pm$ 0.0051
	Mean $\pm$ SD	0.0012 $\pm$ 0.0005	0.0047 $\pm$ 0.0068	0.006 $\pm$ 0.0093	0.006 $\pm$ 0.0046	
Zinc (Zn)	Winter	0.003	0.005	0.007	0.018	0.0083 $\pm$ 0.0067
	Spring	0.031	0.02	0.092	0.035	0.0445 $\pm$ 0.0323
	Summer	0.038	0.041	0.09	0.047	0.054 $\pm$ 0.0242
	Autumn	0.035	0.025	0.013	0.026	0.02475 $\pm$ 0.009
	Mean $\pm$ SD	0.0267 $\pm$ 0.0161	0.0227 $\pm$ 0.0148	0.0505 $\pm$ 0.0468	0.0315 $\pm$ 0.0124	

value 0.008 $\pm$ 0.006 mg/L at site 3, the minimum value 0.003 $\pm$ 0.003 mg/L at site 2. Nonetheless, during the summer and autumn seasons, strong significant differences ($p \leq 0.05$) in Cu values were found between site 3 compared with other sites and seasons.

Concentrations of Cr metal ranged from 0.0001 mg/L at site 2 during the winter to 0.0062 mg/L at site 4 during the summer. The temporal variations reported the highest value 0.0026 $\pm$ 0.0025 mg/L in the summer, the lowest value 0.0002

$\pm$ 0.0005 mg/L in the winter. Whilst, the location variations showed the maximum value 0.0023 $\pm$ 0.0023 mg/L at site 4 and the minimum value 0.0004 $\pm$ 0.0003 mg/L at site 1 (Table 2). The results showed clear significant differences ($p \leq 0.05$) in values of Cr from site 4 during the summer relative to the other sites and seasons.

In cadmium, the concentrations were ranged from 0.0001 mg/L at site 1 in winter to 0.0024 mg/L at site 3 in autumn. The temporal differences through the spring

reported the highest value 0.00142 ± 0.0002 mg/L, the lowest value 0.00023 ± 0.0001 mg/L appeared through the winter. As regards spatial shifts, the maximum value 0.0015 ± 0.0009 mg/L at site 3, and the minimum value 0.0005 ± 0.0005 mg/L at site 1 (**Table 2**). Nonetheless, strong significant differences ($p \leq 0.05$) showed in Cd values between site 1 during the winter relative to the other sites and seasons that recorded the very best levels.

Concentrations of Pb in water samples ranged from 0.001 mg/L at site 1 in winter, summer, and autumn, then at 2 and 3 sites in winter and spring seasons to 0.02 mg/L at site 3 in summer. The seasonal differences showed the highest value 0.011 ± 0.0082 mg/L in summer, the lowest value 0.00125 ± 0.0005 mg/L was recorded in winter. As the spatial variations registered the maximum value 0.006 ± 0.0046 mg/L at site 4, and the minimum value 0.0012 ± 0.0005 mg/L at site 1. Furthermore, the findings showed strong significant differences ($p \leq 0.05$) in Pb values between the 2 and 3 sites during the summer, which reached the very best values compared to the other sites and seasons which tend to be the lowest (**Table 2**).

In Zn the concentrations were varied from 0.003 mg/L at site 1 in winter to 0.092 mg/L at site 3 in spring. Temporal variations observed the highest value 0.054 ± 0.0242 mg/L during the summer and the lowest value 0.0083 ± 0.0067 mg/L during the winter. On the other hand, the position variations showed the maximum value 0.0505 ± 0.0468 mg/L at site 3, the minimum value of 0.0227 ± 0.0148 mg/L at site 2. Accordingly, the statistical analysis showed strong significant differences ($p \leq 0.05$) in Zn values at site 3 during the spring and summer seasons, which gave the highest values compared to other sites and seasons which appear to have the lowest values.

Heavy metals in the sediments

The concentration of studied metals within the sediments was decreasing according to the order following: $\text{Fe} > \text{Ni} > \text{Cd} > \text{Cu} > \text{Cr} > \text{Zn} > \text{Pb}$. During the study period the concentrations of Fe, Ni, and Cd exceeded the permissible limits recommended by [27–29] except Cd, which wasn't detected (ND) during the winter season (**Table 3**). The concentration of Cu exceeded the permissible limits in winter, spring at site 1, in summer and autumn at site 2 consistent with [27], but didn't exceed other standard guidelines set by [28,29], while at sites 3 and 4 it exceeded the permissible limits set by [27,29] during winter, spring and summer, but didn't exceed the other standards established by [28]. The Cr also surpassed the allowable limits at both 1 and 4 sites during the winter,

at both 3 and 4 sites during the autumn. Consistent with all standards, Zn and Pb metals didn't exceed the permissible limits within the sediments (**Table 6**).

The results of this study showed A level of Fe within the sediment ranged from 5,266 mg/kg at site 1 in the summer to 13,960 mg/kg at site 4 in the autumn. The seasonal fluctuations showed the highest value $11,432 \pm 2,408$ mg/kg during the autumn and the lowest value $6,624 \pm 1,251$ mg/kg during the spring. Additionally, the spatial variations found the maximum value $10,608 \pm 3,833$ mg/kg at site 3 and the minimum value $7,909.5 \pm 2,218$ mg/kg at site 2 (**Table 3**). Non-significant differences ($p \leq 0.05$) in Fe values were observed between study sites and seasons consistent with the rules set by [27–29].

Concentration of Ni within the sediments ranged from 36 mg/kg at site 2 during the winter to 103 mg/kg at site 4 during the same season. Thereby, the temporal variations achieved the highest value 71 ± 29 mg/kg in winter and the lowest value 49 ± 12 mg/kg in the spring season. whilst, the situation variations observed the maximum value 74.24 ± 23.27 mg/kg at site 4, the minimum value 47.57 ± 9.991 mg/kg at site 2. The results of the statistical analysis showed no significant differences ($p \leq 0.05$) between the study sites and seasons in the Ni values (**Table 3**).

Copper concentration in sediment samples ranged from 4.6 mg/kg at site 2 during the winter to 33.23 mg/kg at site 4 during the autumn. The seasonal differences found the highest value 24 ± 8.25 mg/kg during the autumn, the lowest value 13 ± 2.65 mg/kg during the spring. Whereas in spatial variations, the maximum value 22.022 ± 8.303 mg/kg was found at site 4, the minimum value 14.267 ± 7.786 mg/kg at site 2. Non-significant variations ($p \leq 0.05$) were observed between the study sites and seasons in the Cu values (**Table 3**).

The concentration of Cr in the sediment samples ranged from 3.06 mg/kg at site 1 during the spring to 47.33 mg/kg at site 4 during the autumn. The seasonal differences showed the highest value 29.3 ± 13.44 mg/kg in autumn, the lowest value 7.39 ± 4.31 mg/kg in springtime. The spatial variations also recorded the maximum value 32.257 ± 16.627 mg/kg at site 4, and the minimum value 10.947 ± 6.875 mg/kg at site 2. Clear significant differences ($p \leq 0.05$) in chromium values were observed at site 4 during the winter and autumn seasons, which reached the highest value relative to the other sites and seasons.

The highest concentration of Cd in the sediment was done 3.98 mg/kg at site 4 during the autumn, whereas non-detected (ND) any concentration at all sites during the winter season.

Table 3 Mean and standard deviation of heavy metals concentrations (mg/kg) in sediments.

Heavy Metal	Season	Site 1	Site 2	Site 3	Site 4	Mean $\pm$ SD
Iron (Fe)	Winter	13,119	6,252	13,934	6,161	9,866 $\pm$ 4,239
	Spring	7,914	5,873	5,276	7,432	6,624 $\pm$ 1,251
	Summer	5,266	9,026	10,475	12,613	9,345 $\pm$ 3,092
	Autumn	8,537	10,483	12,747	13,960	11,432 $\pm$ 2,408
	Mean $\pm$ SD	8,709 $\pm$ 3,264	7909.5 $\pm$ 2218	10,608 $\pm$ 3,833	10,041.5 $\pm$ 3,822	
Nickel (Ni)	Winter	86	36	59.66	103	71 $\pm$ 29
	Spring	66	44.36	37.8	46	49 $\pm$ 12
	Summer	38.8	50.16	59.56	73.8	56 $\pm$ 15
	Autumn	52.73	59.76	66.86	74.16	63 $\pm$ 9.21
	Mean $\pm$ SD	60.88 $\pm$ 20.093	47.57 $\pm$ 9.991	55.97 $\pm$ 12.586	74.24 $\pm$ 23.27	
Copper (Cu)	Winter	18.3	4.6	24	18	16 $\pm$ 8.23
	Spring	16.43	11.56	10.5	14	13 $\pm$ 2.65
	Summer	11.23	21.96	28.46	22.86	21 $\pm$ 7.2
	Autumn	15.2	18.95	28.03	33.23	24 $\pm$ 8.25
	Mean $\pm$ SD	15.29 $\pm$ 2.992	14.267 $\pm$ 7.786	22.747 $\pm$ 8.408	22.022 $\pm$ 8.303	
Chromium (Cr)	Winter	27	7.6	15	45	24 $\pm$ 16.32
	Spring	3.06	5.6	7.7	13.2	7.39 $\pm$ 4.31
	Summer	9.26	9.63	18.36	23.5	15 $\pm$ 6.9
	Autumn	17.36	20.96	31.5	47.33	29.3 $\pm$ 13.44
	Mean $\pm$ SD	14.17 $\pm$ 10.365	10.947 $\pm$ 6.875	18.14 $\pm$ 9.956	32.257 $\pm$ 16.627	
Cadmium (Cd)	Winter	ND	ND	ND	ND	ND
	Spring	1.39	2.16	2.73	3.03	2.33 $\pm$ 0.72
	Summer	1.89	1.24	2.38	3.13	2.16 $\pm$ 0.8
	Autumn	1.81	1.55	2.19	3.98	2.38 $\pm$ 1.1
	Mean $\pm$ SD	1.69 $\pm$ 0.268	1.65 $\pm$ 0.468	2.433 $\pm$ 0.273	3.38 $\pm$ 0.522	
Lead (Pb)	Winter	23	16.3	17.3	17.66	18.565 $\pm$ 3.01
	Spring	16.6	19.13	17.46	21	18.55 $\pm$ 1.94
	Summer	15.4	9.43	10.88	15.13	12.71 $\pm$ 3.01
	Autumn	19.06	20.2	19.96	24.23	20.86 $\pm$ 2.3
	Mean $\pm$ SD	12.515 $\pm$ 3.356	16.265 $\pm$ 4.845	16.4 $\pm$ 3.876	19.505 $\pm$ 3.962	
Zinc (Zn)	Winter	68.33	48	73.33	93	70.66 $\pm$ 18.48
	Spring	55.06	40.25	51.9	47.6	48.70 $\pm$ 6.41
	Summer	74.83	51.96	61.06	68.95	64.2 $\pm$ 9.92
	Autumn	92.1	68.86	74.36	91.93	81.81 $\pm$ 12
	Mean $\pm$ SD	72.58 $\pm$ 15.396	52.267 $\pm$ 12.083	65.162 $\pm$ 10.708	75.37 $\pm$ 21.583	

Table 4 Mean and standard deviation of heavy metal concentrations (mg / kg dry weight) in *C. demersum*.

Season	Fe	Ni	Cu	Cr	Cd	Pb	Zn
Winter	725.93	11.28	2.88	1.91	0.32	1.07	11.57
Spring	326.81	7.35	1.79	0.32	0.64	1.07	8.59
Summer	797.76	12.483	0.601	2.953	0.869	1.483	13.303
Autumn	677.8	10.23	0.89	1.45	0.97	1.25	10.48
Mean	632.075	10.33575	1.54025	1.65825	0.69975	1.21825	10.98575
SD	209.3942	2.193033	1.026697	1.091488	0.288364	0.195837	1.975382
Mean $\pm$ SD	632 $\pm$ 209	10.33 $\pm$ 2.19	1.54 $\pm$ 1.02	1.65 $\pm$ 1.09	0.699 $\pm$ 0.28	1.21 $\pm$ 0.19	10.98 $\pm$ 1.97

Table 5 Mean and standard deviation of heavy metal concentrations (mg / kg dry weight) in *P. pectinatus*.

Season	Fe	Ni	Cu	Cr	Cd	Pb	Zn
Winter	388.72	5.06	1.34	0.24	0.21	0.33	5.75
Spring	419.28	4.72	1.06	0.24	0.81	0.88	7.33
Summer	310.88	3.486	0.253	1.021	0.852	0.441	6.242
Autumn	289.5	4.58	0.76	0.94	0.88	0.78	4.6
Mean	352.095	4.4615	0.85325	0.61025	0.688	0.60775	5.9805
SD	61.83857	0.680849	0.464997	0.428805	0.319962	0.263781	1.132614
Mean $\pm$ SD	352.09 $\pm$ 61.83	4.46 $\pm$ 0.68	0.85 $\pm$ 0.46	0.61 $\pm$ 0.43	0.68 $\pm$ 0.32	0.61 $\pm$ 0.26	5.98 $\pm$ 1.13

Table 6 Permissible limits for concentrations of heavy metals in water, aquatic plants, and sediment.

Guidelines	Iron (Fe)	Nickel (Ni)	Copper (Cu)	Chromium (Cr)	Cadmium (Cd)	Lead (Pb)	Zinc (Zn)	References
Water mg/L	0.3	0.07	2	0.05	0.003	0.01	3	WHO, 2011
	0.3	0.1	1.3	0.05	0.01	0.05	5	EPA, 2002
	0.3	0.1	0.05	0.05	0.005	0.05	0.5	MDPS,1967
Sediment mg/kg	30	16	16	25	0.6	40	110	USEPA,1999
	21	16	35.7	37.5	0.6	35	123	CCME,1999
	30	20	25	25	0.6	40	123	WHO,2004
Aquatic plants mg/kg D W wet	20	10	10	1.3	0.02	2	0.6	WHO,1996

However, the seasonal variations showed the highest value 2.38 ± 1.1 mg/kg during the autumn, and the lowest value 2.16 ± 0.8 mg/kg during the summer. In contrast, the spatial variations detected the maximum value 3.38 ± 0.522 mg/kg at site 4, and the minimum value of 1.65 ± 0.468 mg/kg at site 2. The results appeared clear significant differences ($p \leq 0.05$) in Cd values during the sites and study seasons compared with the winter season, which non-recorded any value in the study sites (**Table 3**).

Lead in the sediment samples has varied between 9.43 mg/kg⁻¹ at site 2 during the summer and 24.23 mg/kg at site 4 during the autumn. Nevertheless, the temporal differences found the highest value 20.8625 ± 2.3 mg/kg during the autumn, and the lowest value 12.71 ± 3.01 mg/kg through the summer. Whilst, the spatial differences proved the maximum value 19.505 ± 3.962 mg/kg at site 4, and the minimum value 12.515 ± 3.356 mg/kg at site 1. Results of the statistical analysis showed non-significant differences ($p \leq 0.05$) in Pb values between study sites and seasons (**Table 3**).

The finding of examined Zn in the sediment samples varied from 40.25 mg/kg at site 2 during the spring to 93 mg/kg at site 4 during the winter. The differences due to the season showed the very best 81.81 ± 12 mg/kg during the autumn while the lowest value was 64.2 ± 9.92 mg/kg during the summer. As regards spatial shifts, the maximum value

75.37 ± 21.583 mg/kg at site 4, the minimum value 52.267 ± 12.083 mg/kg at site 2. Thereby, the statistical analysis demonstrated non-significant differences ($p \leq 0.05$) in Zn values between the study sites and seasons (**Table 3**).

Heavy metals in aquatic plants

This study was conducted to estimate seven heavy metals within the Al-Hawizeh marsh on a seasonal basis for 2 species of submerged aquatic plants as *Ceratophyllum demersum* Linn and *Potamogeton pectinatus* Lam. Thereby, the (Mean $\pm$ SD) for heavy metals content in examined plants were shown within **Tables 4 and 5**.

Iron concentration at the *C. demersum* plant varied from 326.81 mg/kg dry weight during the spring to 797.76 mg/kg dry weight during the summer. However, the Mean $\pm$ SD of Fe value reached 632 ± 209 mg/kg dry weight in this species (**Table 4**). Nickel concentration in this plant came between 7.35 mg/kg dry weight during the spring and 12.483 mg/kg dry weight during the summer. Thereby, Ni achieved 10.33 ± 2.19 mg/kg dry weight. The concentration of Cu varied from 0.601 mg/kg dry weight during the summer to 2.88 mg/kg dry weight during the winter. The Cu metal getting to 1.54 ± 1.02 mg/kg dry weight. In Cr metal, the concentrations ranged from 0.32 mg/kg dry weight during the spring to

2.935 mg/kg dry weight during the summer. Nonetheless, the Cr reached 1.65 ± 1.09 mg/kg dry weight. Cadmium concentration varied from 0.32 mg/kg dry weight during the winter to 0.97 mg/kg dry weight during the autumn. Moreover, the Cd reached 0.699 ± 0.28 mg/kg dry weight. The value of Pb concentration varied between 1.07 mg/kg dry weight during the winter and spring seasons to 1.483 mg/kg dry weight during the summer. As well as, the Pb reported 1.21 ± 0.19 mg/kg dry weight. The concentration of Zn laid between 8.59 mg/kg dry weight during the spring to 13.303 mg/kg dry weight during the summer. Also, the Zn recorded 10.98 ± 1.97 mg/kg dry weight.

Overall, the results from the metals examined in the *C. demersum* plant declined as follows: Fe > Zn > Cd > Cr > Ni > Pb > Cu. Nonetheless, the concentrations of Fe, Cd, Zn, and Cr metals exceeded the allowable limits set by [30] in all study seasons. Nickel metal was within the permissible limits, while both Cu and Pb metals did not reach the permissible limits in all sample seasons during the present study (Table 4).

In *P. pectinatus* the heavy metals were accumulated of varying concentrations at the tested sites. Fe ranged from 289.5 mg/kg dry weight during the spring to 419.28 mg/kg dry weight during the autumn. Thereby, the Iron value achieved 352.09 ± 61.83 mg/kg dry weight (Table 5).

The Ni concentration ranged from 3.486 mg/kg dry weight during the summertime to 5.06 mg/kg dry weight during the winter. Thereby, the Ni getting to 4.46 ± 0.68 mg/kg dry weight. Concentration of Cu differs from 0.253 mg/kg dry weight during the summer to 1.34 mg/kg dry weight during the winter. As well as, the value of Cu had 0.85 ± 0.46 mg/kg dry weight. In Cr the concentrations varied from 0.24 mg/kg dry weight during the winter and spring seasons to 1.021 mg/kg dry weight during the summer. However, the value of Cr gave 0.61 ± 0.43 mg/kg dry weight. Cadmium concentration in this plant was varied between 0.21 mg/kg dry weight during the winter and 0.88 mg/kg dry weight during the autumn. Thereby, the value of Cd gave 0.69 ± 0.32 mg/kg dry weight. Lead concentrations ranged from 0.33 mg/kg dry weight during the winter to 0.88 mg/kg dry weight during the spring. Nevertheless, the value of Pb reached 0.61 ± 0.26 mg/kg dry weight. Value of Zn concentration laid between 4.6 mg/kg dry weight during the autumn to 7.33 mg/kg dry weight during the spring. Further, the value of Zn had 5.98 ± 1.13 mg/kg dry weight.

Generally, the results of testing these metals in the *P. pectinatus* plant decreased within the following: Fe > Zn > Cd > Ni > Cr > Pb > Cu. Therefore, the concentrations of Fe, Cd,

and Zn metals exceeded the permissible limits set by [30] in all seasons during the present study. Whereas, Ni, Cu, Cr, and Pb did not exceed the permissible limits in all seasons during the current study. Moreover, all permissible limits for the concentration of heavy metals in water, sediment, and aquatic plants are illustrated in (Table 6).

Distribution of heavy metals among water, sediment, and aquatic plants

Pearson's analysis for test (r) value was performed at significance levels $p \leq 0.05$ to determine the correlation of heavy metals with the samples. The results showed correlations of metals between water and sediments are very weakly positive significant correlation for Ni ($r = 0.331$ *), also a weakly positive significant correlation between water and plant for Zn ($r = 0.418$ *), Ni ($r = 0.406$ *) and Fe ($r = 0.388$ *). Whilst has shown a good positive significant correlation between sediment and plant for Fe ($r = 0.931$ *), Zn ($r = 0.924$ *), Ni ($r = 0.883$ *), Pb ($r = 0.744$ *). Additionally, non-significant correlations were reported for other values among these three environments. However, the results for water, sediment, and plant correlation coefficients are summarized in Table 7.

DISCUSSION

The variation in concentrations content of heavy metal could be attributed to interactions between multiple factors that affect dissolved metal concentrations, like phytoplankton and aquatic plant densities that absorbed or adsorbed ionic metals, also because the emissions of fuel-burning within the marsh during the summer season [31]. The existence of heavy metals in aquatic environments has created serious

Table 7 Pearson Correlation Coefficient (r) values for Fe, Cu, Cr, Ni, Zn, Pb, and Cd between Water/ Sediment; Water /Plant and Sediment/Plant.

Element	Correlation Coefficient (r^2)		
	Water/ Sediment	Water/ Plant	Sediment/ Plant
Fe	0.274	0.388*	0.931*
Cu	0.229	0.302	0.735
Cr	0.179	0.254	0.626
Ni	0.331*	0.406*	0.883*
Zn	0.309	0.418*	0.924*
Pb	0.284	0.279	0.744*
Cd	0.098	0.255	0.553

* Correlation is significant at $p \leq 0.05$ level.

concerns about their effect on plant and animal life [32]. The increase in the quantity of these metals within the environment has dramatically in recent decades as a consequence of anthropogenic behavior. On the opposite hand, the rise of heavy metals in marsh water and sediment could also be due to the very fact this area receives large quantities of waste, agricultural run-off, and toxic effluents from the Tigris River [33], whilst the seasonal differences in concentrations of metals at different sites are often attributed to changes within the quantity of agricultural run-off and waste effluents discharged into the Marsh [34].

The results of this study revealed the Fe metal was the heaviest metal abundant from the varied sites of the marsh. Thereby, the Fe concentrations in water samples were below the permissible limit, while such content in sediments and plants was over the permissible limit in altogether studied sites during the present period. Iron is involved, directly or indirectly, in many life plant processes: chlorophyll biosynthesis, photosynthesis, respiration, elemental nitrogen fixation, reduction of nitrate and nitrite, carbohydrate metabolism, and redox in the various systems. As a result, the high concentrations of this metal can cause plant oxidizing stress [35].

Zinc is a crucial micronutrient for all organisms because it forms a part of the active site in the various metalloenzymes. Also, its concentration in the water and sediment samples did not exceed the permissible limit in all locations during study time (Tables 2 and 3). Zinc plays a crucial role in protein synthesis and maybe a metal that shows a fairly low concentration in surface water due to its restricted mobility from the place of rock weathering or the natural sources. Furthermore, the concentrations of Zn in *C. demersum* and *P. pectinatus* plants were high in all sites through study seasons, which might be due to the growth of these macrophytes which possesses a unique capacity to absorb a large amount of zinc during their growing periods or photosynthetic assimilation as reported by several researchers [36].

Copper is one of the essential elements required for the numerous plant enzymatic activities. Windblown dust releases copper particulates into the atmosphere; volcanic eruptions; and anthropogenic causes. During this study, copper concentration in both water and aquatic plant samples was less than the suitable level, which may be attributable to macrophyte bioaccumulation also because of the lower surface run-off from nearby farming, planting fields, and sewage settlements containing large quantities of dissolved organic matter, which promotes the mobility of copper [37].

Likewise, copper fate in water is complex and determined

by pH, dissolved oxygen, and therefore the presence of oxidizing agents and chelating compounds or ions. The finding showed the concentration of copper in sediments was above the permissible limit at sites 1, 3, and 4 during the winter, at sites 2, 3, and 4 during summer and autumn respectively. Although it didn't exceed the permissible limit in spring except at site, On the other hand, the *C. demersum* plant was one among the foremost tolerant macrophytes against the copper [38]. Therefore, they recommended it as a reliable bio-indicator in freshwater for medium to high levels of copper.

Chromium could also be a metal that's rare extremely. Its frequency and quantities are typically very low within the marine environments. Cr isn't important for plants, it belongs to the category of harmful elements also, it doesn't accumulate in large quantities within plants [39]. The marshy plants are known to absorb and accumulate heavy metals from sediments and polluted water. During this research, the chromium content in water samples didn't exceed the permissible limits at all sites through the study seasons (Table 2), which may be due to its low solubility, whereas it had the highest bioaccumulation potential in the sediments, especially at site 1 in winter and site 4 in winter and autumn (Table 3), whereas in aquatic plants like *C. demersum* has exceeded the permissible limit at all sites during the study period (Table 4).

Pb metal isn't a crucial element for the expansion of plants but it's absorbed with other elements by the plant [40]. it's considered a toxic metal that accumulates by ingestion by food, water, and inhalation in plant and animal tissues. The key source of lead in water might be also from toxic industrial waste, from mixing gun powder in soil, waste batteries into aquatic environments which causes deposition of lead. The results detected the concentrations of lead in water, soil, and plant samples (*C. demersum* and *P. pectinatus*) didn't exceed allowable limits during the present study (Tables 2, 3, 4, and 5), except the sites 2 and 3 which observed a minor variance in lead value in water samples.

In sediment samples, the cadmium exceeded the allowable limit at all sites during the spring, summer, and autumn seasons, but didn't record any value at all sites during the winter, which might be due to the variations in the quantity of agricultural run-off and discharged sewage effluent [34,35].

Recently, Ni has been recognized as one of the elements that essential for higher plants because it is represented as a component of enzymes critical for normal life processes. Nickel in water samples was below the allowable limit. Considering nickel in *C. demersum* plant was above the recommended limit set by [26] except for the spring season, also

the *P. pectinatus* plant was below the recommended limits. During the study period, the concentration of nickel-metal in sediment samples was above the acceptable limit, which may be attributed to the fact this marsh receives massive amounts of sewage, agricultural runoff, and toxic effluents from the Tigris River and its branches [33].

The concentrations of Fe and Cd in the *C. demersum* and *P. pectinatus* plants were highest compared to other metals, while Cu was the lowest concentration in these species, close to the distribution pattern of metal concentrations in sediments, suggesting that the concentration of metals in plants is correlated to a point with the concentration of sediments, these results almost like those of previous studies [41].

The findings showed that the interior transportation of metals also apparently differs between the plant species and therefore the metal type. This study suggests that plant uptake of metals varied with the various plant species. However, the higher concentrations of metal in *C. demersum* for Fe, Cd, Zn, Ni, and Cr indicate greater potential for *C. demersum* to absorb these sediment metals than that of *P. pectinatus*.

The highest concentrations of heavy metals were present in water during the summer, while in sediments during the autumn, and this may be due to the death and decay of the macrophytes that release them, elements in-media were locked up. In contrast, the lowest concentrations of heavy metals in water found during the winter season and the lowest concentrations present in sediments during the spring season may be due to the absorption or oxidation of elements by the macrophytes during their growth time, sequestering more elements from the growing media.

Overall, the distribution of heavy metals in sediment was $Fe > Ni > Cd > Cu > Cr > Zn > Pb$. For plants, as order following $Fe > Zn > Cd > Cr > Ni > Pb > Cu$, whereas in water was within the sequence $Fe > Ni > Zn > Pb > Cr > Cd$.

CONCLUSIONS

The results showed all metals in water were below the permissible limits. Metals of Fe, Ni, and Cd have exceeded the permissible limits, whereas, Zn and Pb did not exceed the recommended limits in the sediment samples. When examined samples of *C. demersum* plant showed Fe, Cd, Zn, and Cr have exceeded the allowable limits, but Cu and Pb did not exceed the permissible limits, also in *P. pectinatus* plant Fe, Cd, and Zn have exceeded the permissible limits, while Ni, Cu, Cr, and Pb did not exceed the permissible limits in all seasons during the present study. Therefore, we conclude that heavy metals accumulated in the water at lower rates than

what is found in plants and sediments, respectively, and there are self-purification processes for the water marsh conducted by submerged plants for those heavy metals. Additionally, the sediments collected from the study areas contained high percentages of heavy metals, because the main reservoir for those metals needs a susceptibility to sedimentation and thus their weighting density allows them to settle at the bottom of the marsh.

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