

Heavy Metals Levels in the Al-Shamiyah River: A Lotic Ecosystem Case Study

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ABSTRACT

Monitoring lotic ecosystems is vital for addressing sustainability issues. The Al-Shamiyah River is the primary source of water for various daily activities in the Al-Shamiyah district. This study assessed the pollution levels of the river by measuring the concentration and distribution of heavy metals—specifically chromium, cadmium, manganese, copper, zinc, and lead—in both the river's water and sediments. The concentrations of heavy metals in the water ranged from 0.05 to 1.44µg/ L for copper (Cu), 1.57 to 7.25µg/ L for manganese (Mn), 0 to 1.7µg/ L for cadmium (Cd), 0.02 to 1.33µg/ L for lead (Pb), 0.08 to 2.74µg/ L for zinc (Zn), and 0.44 to 1.84µg/ L for chromium (Cr). In the particulate phase, the concentrations ranged from 1.5 to 35.44µg/ g for copper (Cu), 56.16 to 284.49µg/ g for manganese (Mn), 1.50 to 10.72µg/ g for cadmium (Cd), 0.25 to 7.73µg/ g for lead (Pb), 78.71 to 717.76µg/ g for zinc (Zn), and 37.64 to 410.92µg/ g for chromium (Cr). The concentration of dissolved heavy metals in the water was lower than that in the particulate phase. In the sediments, the exchangeable phase contained the following order of concentrations: Mn > Zn > Cr > Pb > Cu > Cd, while the residual phase showed the following order: Zn > Mn > Cr > Pb > Cu > Cd. The concentration of heavy metals in the exchangeable phase was lower than in the residual phase. This study enhances our understanding of the impact of anthropogenic activities on the river's health. The findings highlighted the importance of protecting the river from the influx of heavy metals due to surrounding activities, in order to ensure its long-term sustainability.

INTRODUCTION

Heavy elements (HM) exist naturally in different ecosystems with various concentrations (Ezeronye & Ubalua, 2005; Al-Sareji *et al.*, 2021). Heavy elements are essential because they are linked to growth, development, and reproduction processes such as Mn, Cu, and Zn. Ahmed *et al.* (2015) explained that heavy metals are toxic if they exceed permissible limits. Heavy elements affect the water ecosystem dynamics according to their phases in water and sediment (Linnik & Zubenko, 2000).

Heavy metals are toxic to humans if they enter the body through inhalation or swallowing, and they accumulate in the body with drinking water and lead to poisoning (Wang *et al.*, 2013; Al-Obeidi & Al-Jumaily, 2020; Salman *et al.*, 2023). The increase in HMs in the river's aquatic environment results from human activity, which works to discharge pollutants into the river, such as agricultural pollutants. Fertilizers and pesticides are used to prepare agricultural fertilizers (Brodin *et al.*, 2017; Al-Jumaily & Al-Berzanje, 2020).

Al-Khafaji *et al.* (2011) found during a survey of the Gharraf River in Nasiriyah, Iraq, that the concentration of the three HMs, lead, cadmium, and copper, was high in the river water. Hassan *et al.* (2010) found the lead concentration was low in the Euphrates River, while the concentration of copper in this river was high. Additionally, the study by Al-Asadi and Al-Kafari (2022) on the waters of the Al-Diwaniyah River found that the concentrations of heavy metals (manganese, nickel, lead, zinc, chromium, copper, arsenic, and cadmium) were generally low in the river water, except for lead. However, the concentrations of these metals were higher in the river sediments, although they remained within permissible limits.

Al-Ghanimy and Al-Rekabi (2023) found that concentrations of HMs (dissolved phase) in the Euphrates River water were lower than their concentration in the particulate phase, as cadmium, lead, and zinc were higher in the particulate phase than from the other metals under study. Additionally, Saber *et al.* (2023) found that the concentrations of lead pose great environmental risks compared to the risks of the HMs (chromium, copper, and zinc) in the sediments of this river. Additionally, high concentrations of chromium, zinc, and manganese were recorded in the sediments of the Diyala River. In contrast, the concentration of copper was low, and it was considered free of lead contamination (Mijbas *et al.*, 2024). Salman and Al-Shammari (2020) evaluated the water quality of the Hilla River by studying some characteristics, and they estimated the concentrations of some HMs in the Musayyib River on the Euphrates River and monitored their transport in water and sediments. On the other hand, a recent study focused on water quality evaluation for some Iraqi rivers, and found that some rivers are carbon dioxide saturated (Kadhim & Hamdan, 2024).

Based on toxic cases of fish and reduced aquatic plant distribution in Al-Shamiyah River, southern Iraq, and the little available literature about heavy metal in this river, we hypothesized that HM concentrations are high in the river.

MATERIALS AND METHODS

1. The description of study area

Euphrates River enters Iraqi territory and continues to flow there for a distance of 150km when it reaches the Hindiyah area, with the presence of the Hindiyah Dam, the Euphrates River branches into its two main branches, which are the Hilla River and the Hindiyah River (WHO-Unicef-2000). Then the Euphrates River (Al-Hindiyya River)

branches into two branches: the Kufa River and Al-Shamiyah River, where Al-Shamiyah River, which is the current study area, continues in the Abbasiyah, Al-Hurriya, Al-Salhiya, Al-Shamiyah, and Ghamas districts until it connects with the Euphrates River in Al-Shinafiya District in Al-Qadisiyah Governorate.

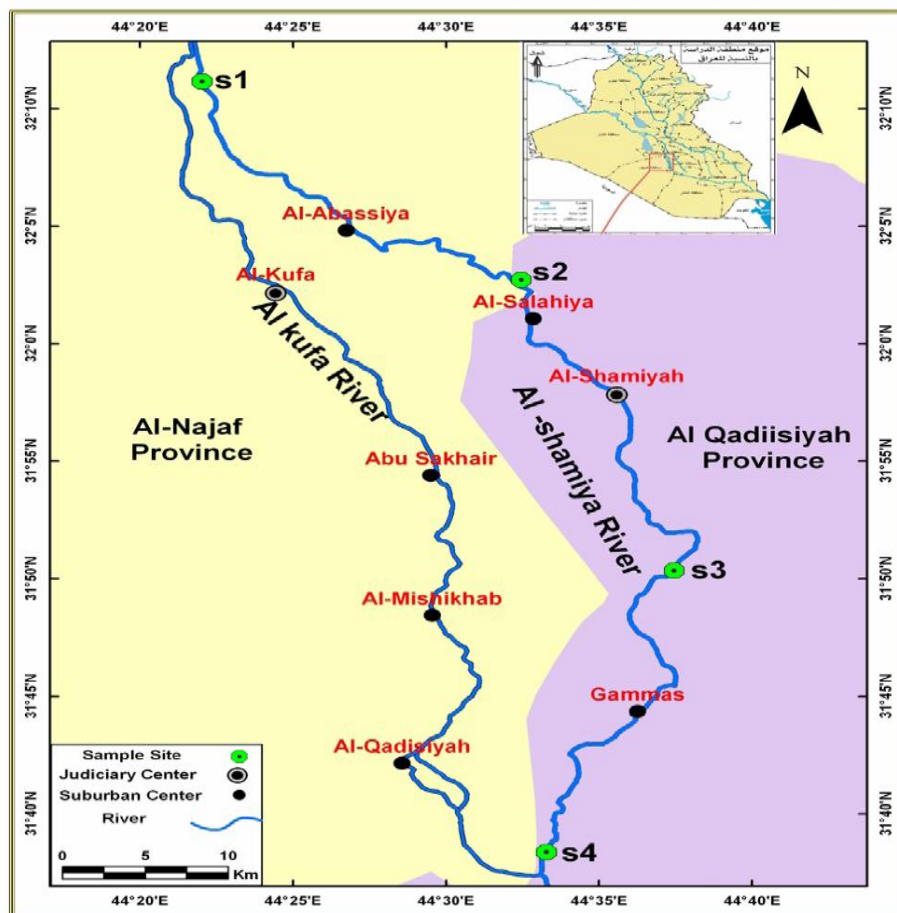


Fig. 1. The studied sites on Al-Shamiyah River

Al-Shamiyah River enters the lands of Al-Qadisiyah Governorate from the northwestern side of the governorate within the lands of Al-Shamiyah District. It enters the western borders of Al-Mahnawiyah District, heading toward the south, passing through Al-Saliyah District, the center of Al-Shamiyah District, and Ghamas District. Its length is 125km, and its average drainage capacity reached $180\text{m}^3/\text{s}$. It is the main source of irrigation in these areas, and the land irrigated by it reaches 384,000 dunams. Samples were collected monthly during the study period from March 2023 until March 2024 from the study sites that were identified using a geographical site system device according to each site (Fig. 1). Site 1 is located at longitude 44.3668°E and latitude 32.1853°N ; Site 2 at longitude 44.5383°E and latitude 32.0445°N ; Site 3 at longitude 44.6240°E and latitude 31.8392°N ; and Site 4 at longitude 44.5539°E and latitude 31.6392°N .

2. Water samples

Water samples were taken from 30cm depth using plastic containers with a capacity of 5 liters to conduct tests for heavy metals during the study period from March 2023 until March 2024 from Al-Shamiyah River and were preserved in special polyethylene containers. Heavy metal ions, dissolved heavy metals, and particulate heavy metals were addressed as follows:

2.1. Heavy metals extraction from water

2.1.1. Dissolved phase

Water samples were transported from the study sites to the laboratory. To separate the dissolved phase from the particulate phase, two liters of water from each sample (three replicates per site) were filtered through 0.45 μ m Millipore filter paper. The filter paper was pre-weighed after being washed with dilute nitric acid (0.5 N), followed by distilled water, and dried at 60°C for 12 days.

The filtered water samples were then concentrated by passing them through an ion exchange column (50 \times 2cm) containing Chelex-100 resin in the sodium form (50-100 mesh), supplied by Bio-Rad, at a flow rate not exceeding 5ml/ min. Heavy metal ions were subsequently washed using 80ml of dilute nitric acid (2 N), and the solution was evaporated at 70°C until dry. After evaporation, 1ml of concentrated nitric acid (HNO₃) and 10ml of deionized water were added, and the solution was allowed to dissolve completely (Riley & Taylor, 1968).

Finally, the volume was adjusted to 25ml with deionized distilled water and stored in polyethylene bottles until the concentrations of elemental ions were measured using flame atomic absorption spectrometry. The results were expressed in micrograms per liter (APHA, 2003).

2.1.2. Particulate phase

The filter papers used to filter the water samples were dried at 80°C for 48 hours and then weighed to determine the amount of plankton and particulate heavy metals removed. To extract the ions from the particulate matter, 0.5 grams of the dried samples were placed in special Teflon containers and treated with 6 ml of a 1:1 mixture of HCl and HNO₃. The mixture was heated at 80°C until nearly dry. Then, 4ml of a 1:1 mixture of concentrated perchloric acid (HClO₄) and hydrofluoric acid (HF) was added, and the mixture was evaporated to a near-dry state.

The residue was dissolved with 20 ml of diluted hydrochloric acid (0.5 N) and allowed to stand for ten minutes. The sample was then centrifuged for 20 minutes at 3000 rpm. The supernatant was transferred to a 25ml volumetric flask. The sediment was washed with ion-free distilled water, and the washing water was added to the volumetric flask, bringing the total volume to 25ml (Sturgeon *et al.*, 1982).

The samples were stored in polyethylene containers until analyzed using flame atomic absorption spectrometry.

3. Collection of sediment sample

Sediment samples were collected from the Al-Shamiyah River in the study locations between March 2023 and March 2024. The samples were obtained using a Crap device and placed in clean plastic bags for transport to the laboratory. Once in the laboratory, the sediment samples were mixed thoroughly, and the solids were removed. The samples were then dried at 60°C for 48 hours.

After drying, the sediments were ground in a ceramic mortar and sieved through a nylon sieve with a 65-micrometer mesh diameter. The samples were then stored in labeled polyethylene containers until the heavy metal ions were extracted, including both the exchangeable fraction and the remaining residual fraction, as follows:

3.1. Heavy metals extraction from sediment

3.1.1 Exchangeable phase

Chester and Voutsinou's (1981) method was used to extract HM ions in the exchangeable portion of the sediment. We put one gram of the sample in a 50ml test tube. Then, 20ml of 0.5 N hydrochloric acid was added, placed for 16h in a shaking device, and separated in a centrifuge at 3000rpm for 20 minutes. The solution was then transferred to polyethylene bottles and was stored until measured by a flame atomic absorption spectrophotometer.

3.1.2 Residual phase

After the exchanged portion was extracted from the sediment, the remaining sediment portion was taken and was digested to extract the remaining element ions in the sediment based on **Sturgeon et al. (1982)** as follows:

Forty milliliters of distilled, ion-free water were added to the sediment to remove traces of the exchanged elements and the acids used. The samples were then centrifuged at 3000 rpm for 20 minutes to separate the wash water. The sediment was quantitatively transferred to a Teflon beaker, taking care to avoid losing any precipitate. The test tube was rinsed with distilled, ion-free water to remove any residue stuck to the walls. The washing water was added to the beaker containing the sediment sample.

The sample was evaporated at 80°C, and 6ml of a 1:1 mixture of concentrated nitric acid (HNO₃) and hydrochloric acid (HCl) was added to the residue. This mixture was evaporated again at 80°C until dry. Next, 4ml of a 1:1 mixture of hydrofluoric acid (HF) and perchloric acid (HClO₄) was added, and the solution was evaporated until dry.

The residue was then dissolved in 20 ml of 0.5 N hydrochloric acid (HCl) and allowed to stand for 10 minutes. The solution was centrifuged for 20 minutes at 3000 rpm. The supernatant was transferred to a 25ml volumetric flask. The sediment was

washed with deionized water, and the wash water was added to the volumetric flask. The total volume was then adjusted to 25ml, and the solution was stored in clean polyethylene bottles until analysis by flame atomic absorption spectrometry.

4. Quality assurance and quality control (QA/QC)

Water and sediment samples from the Al-Shamiyah River were analyzed in the Environmental Laboratory, Department of Life Sciences, at the College of Education, University of Al-Qadisiyah, and the Biochemistry Laboratory, College of Pharmacy, University of Kufa, Iraq.

The integrity of the Quality Assurance and Quality Control (QA/QC) process for the sample analysis was maintained by following standardized procedures and using blank and standard solutions for heavy metals (BDH from UAE). A total of five replicates of each element, using both standard and blank solutions, were analyzed. All standard solutions were of high quality and accuracy. To minimize random error, samples were carefully measured, and three replicates were performed for each sample to determine the concentration of each element. The final value was the average of the three measurements. The recovery rates for metals in the standard reference material ranged between 99.22 and 99.92% for each element measured.

RESULTS

1. Heavy metals in water

1- Copper (Cu)

Copper concentrations exhibited seasonal variation in the dissolved phase. The lowest concentration was recorded at 0.05 $\mu\text{g}/\text{L}$ in spring 2023 at site 3, while the highest concentration was 1.44 $\mu\text{g}/\text{L}$ in winter 2024 at site 1.

In the particulate phase, the lowest copper concentration was 1.5 $\mu\text{g}/\text{g}$ in spring 2024 at site 1, whereas the highest concentration, 35.44 $\mu\text{g}/\text{g}$ dry weight, was recorded in spring 2023 at the same site (Table 1 & Fig. 2). Statistical analysis revealed significant differences ($P < 0.05$) between seasons and study sites for both the dissolved and particulate phases.

2- Manganese (Mn)

Manganese concentrations in the dissolved phase showed seasonal variations. The lowest concentration, 1.57 $\mu\text{g}/\text{L}$, was recorded in the spring of 2023 at site 3, while the highest concentration, 7.25 $\mu\text{g}/\text{L}$, was recorded in the summer of 2023 at site 4.

In the particulate phase, the lowest concentration, 56.16 $\mu\text{g}/\text{g}$, was recorded in the spring of 2023 at site 4, and the highest concentration, 284.49 $\mu\text{g}/\text{g}$, was observed in the summer of 2023 at site 1 (Table 1 & Fig. 2). Statistical analysis revealed significant

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differences ($P < 0.05$) between seasons and study sites for both the dissolved and particulate phases.

3- Cadmium (Cd)

The lowest concentration of cadmium in the dissolved phase was recorded as imperceptible in the spring of 2024 at site 4, while the highest concentration, 1.70 $\mu\text{g/L}$, was observed in the summer of 2023 at site 1.

In the particulate phase, the lowest concentration, 1.5 $\mu\text{g/g}$ (by weight), was recorded in the winter of 2024 at site 4, while the highest concentration, 10.72 $\mu\text{g/g}$ dry weight, was recorded in the spring of 2023 at site 1 (Table 1 & Fig. 3). Statistical analysis showed significant differences ($P < 0.05$) between the seasons and study sites for both the dissolved and particulate phases.

4 – Lead (Pb)

The lowest concentration of lead in the dissolved phase was observed at 0.02 $\mu\text{g/L}$ at site 1 in autumn 2023 and in spring 2024 at site 3. The highest concentration, 1.33 $\mu\text{g/L}$, was recorded in the summer of 2023 at site 2.

In the particulate phase, lead concentrations exhibited seasonal variations. The lowest concentration, 0.25 $\mu\text{g/g}$ dry weight, was recorded at site 1 in spring 2023, while the highest concentration, 7.73 $\mu\text{g/g}$ dry weight, was observed at site 4 in the same season (Table 1 & Fig. 3). Statistical analysis revealed significant differences ($P < 0.05$) between seasons and study sites for both the dissolved and particulate phases.

Table 1. Average concentration of heavy metals in water, dissolved phase ($\mu\text{g/L}$), particulate phase ($\mu\text{g/g}$ dry weight) in Al-Shamiyah River

| Sites | | | 1 | | | | 2 | | | | | |
|--------|---------|---------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|
| Metals | Samples | Seasons | Spring 2023 | Summer 2023 | Autumn 2023 | Winter 2024 | Spring 2024 | Spring 2023 | Summer 2023 | Autumn 2023 | Winter 2024 | Spring 2024 |
| Cu | Water | Dissol. | 0.61 | 1.38 | 0.46 | 1.44 | 1.43 | 0.38 | 0.42 | 0.54 | 1.22 | 1.32 |
| | | Parti. | 35.44 | 14.59 | 21.26 | 8.06 | 1.5 | 8.94 | 16.61 | 13.08 | 3.6 | 5.26 |
| Mn | Water | Dissol. | 2.31 | 3.99 | 5.18 | 5.65 | 3.23 | 2.07 | 4.11 | 3.56 | 5.39 | 3.85 |
| | | Parti.. | 171.14 | 284.49 | 183.99 | 176.49 | 169 | 177.48 | 168.07 | 152.75 | 176.94 | 172.21 |
| Cd | Water | Dissol. | 0.18 | 1.7 | 0.3 | 0.04 | 0.09 | 0.17 | 0.31 | 0.05 | 0.08 | 0.02 |
| | | Parti.. | 10.72 | 5.05 | 3.33 | 6.78 | 6.78 | 1.6 | 3.46 | 3.29 | 2.8 | 2.56 |
| Pb | Water | Dissol. | 0.83 | 0.61 | 0.02 | 0.11 | 0.02 | 0.44 | 1.33 | 0.06 | 0.15 | 0.12 |
| | | Parti.. | 0.25 | 3.95 | 2.27 | 1.39 | 2.16 | 2.67 | 4.12 | 2.68 | 2.88 | 2.62 |
| Zn | Water | Dissol. | 0.08 | 2.74 | 0.37 | 1.26 | 1.47 | 0.55 | 0.88 | 0.35 | 1.24 | 2 |
| | | Parti.. | 361.79 | 471.6 | 717.76 | 272.24 | 563.96 | 331.45 | 337.06 | 307.69 | 78.71 | 105.16 |
| Cr | Water | Dissol. | 0.5 | 1.84 | 1.39 | 1.05 | 1.04 | 0.44 | 1.65 | 1.59 | 0.89 | 1.33 |
| | | Parti.. | 65.36 | 37.64 | 91.18 | 168.22 | 210.03 | 121.61 | 145.11 | 97.46 | 179.95 | 233.26 |
| | | | 3 | | | | 4 | | | | | |

| Metals | samples | seasons | Spring 2023 | Summer 2023 | Autumn 2023 | Winter 2024 | Spring 2024 | Spring 2023 | Summer 2023 | Autumn 2023 | Winter 2024 | Spring 2024 |
|--------|---------|---------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|
| Cu | Water | Dissol. | 0.05 | 0.36 | 0.6 | 1.06 | 1.37 | 0.42 | 0.44 | 0.65 | 1.42 | 1.42 |
| | | Parti.. | 12.29 | 13.39 | 9.57 | 4.97 | 3.75 | 4.08 | 4.91 | 10.57 | 3.98 | 4.96 |
| Mn | Water | Dissol. | 1.57 | 4.05 | 3.44 | 5.25 | 3.54 | 2.99 | 7.25 | 5.25 | 6.77 | 5.66 |
| | | Parti.. | 86.34 | 109.2 | 118.1 | 117.11 | 103.04 | 56.16 | 69.46 | 75.92 | 66.62 | 57.58 |
| Cd | Water | Dissol. | 0.29 | 0.8 | 0.04 | 0.05 | 0.01 | 0.46 | 0.33 | 0.38 | 0.07 | 0 |
| | | Parti.. | 3.83 | 4.59 | 5 | 1.67 | 2.96 | 3.3 | 4.15 | 1.66 | 1.5 | 6.67 |
| Pb | Water | Dissol. | 0.58 | 1.28 | 0.14 | 0.16 | 0.02 | 0.69 | 0.16 | 0.67 | 0.09 | 0.04 |
| | | Parti.. | 0.69 | 2.3 | 1.41 | 1.55 | 1.28 | 7.73 | 2.43 | 1.72 | 1.48 | 1.22 |
| Zn | Water | Dissol. | 0.19 | 1.44 | 0.43 | 1.65 | 0.38 | 0.91 | 0.71 | 0.33 | 0.87 | 0.36 |
| | | Parti.. | 184.7 | 483.56 | 276.14 | 187.83 | 199.95 | 387.93 | 461.86 | 115.15 | 353.52 | 139.14 |
| Cr | Water | Dissol. | 0.57 | 1.46 | 0.9 | 0.94 | 0.94 | 0.5 | 1.7 | 0.65 | 1.19 | 1.06 |
| | | Parti.. | 57.3 | 117.88 | 410.92 | 204.29 | 221.78 | 286.11 | 92.77 | 64.81 | 151.86 | 209.38 |

Dissol. = Dissolved, Parti.= particulate phase

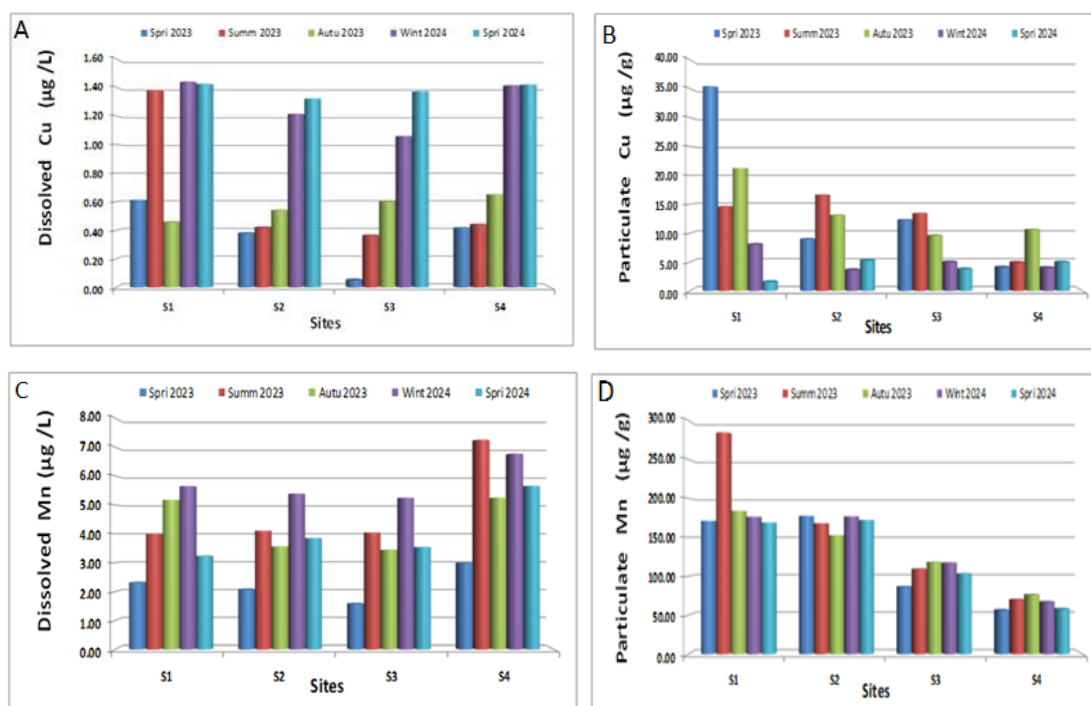


Fig. 2. Seasonal changes of the dissolved and particulate phases of copper (Cu) **A** and **B**, respectively. The dissolved and particulate phases of manganese (Mn) concentrations **C** and **D**, respectively, were in the water of Al-Shamiyah River in the four sites during the study period

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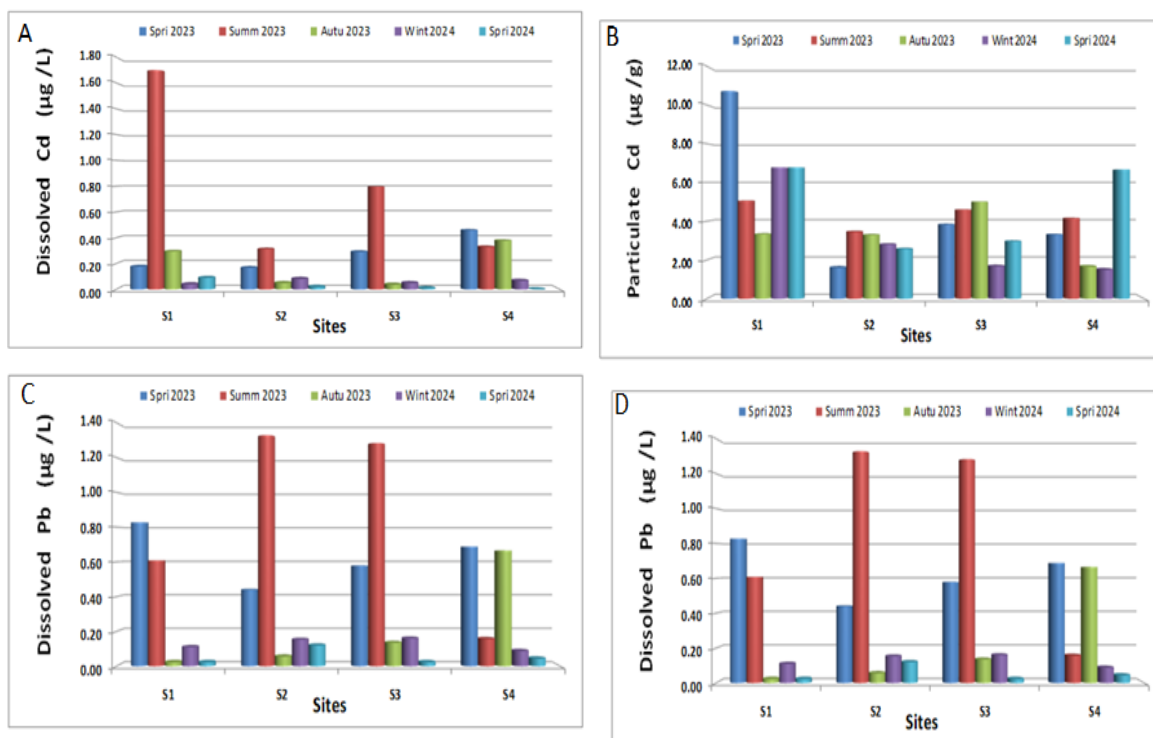


Fig. 3. Seasonal changes in the dissolved phase (A) and particulate phase (B) of cadmium (Cd), and in the dissolved phase (C) and particulate phase (D) of lead (Pb) in the water of the Al-Shamiyah River at the four sites during the study period

5 - Zinc (Zn)

Zinc concentrations in the dissolved phase of the Al-Shamiyah River showed seasonal variation. The lowest concentration, $0.08\mu\text{g/L}$, was recorded at site 1 in the spring of 2023, while the highest concentration, $14.02\mu\text{g/L}$, was observed in the summer of 2023 at site 2.

In the particulate phase, the lowest concentration, $78.71\mu\text{g/g}$ dry weight, was recorded at site 2 in winter 2024. The highest concentration, $717.76\mu\text{g/g}$ dry weight, was observed in autumn 2023 at site 1 (Table 1 & Fig. 4). Statistical analysis revealed significant differences ($P \leq 0.05$) between seasons and study sites for both the dissolved and particulate phases.

6 – Chromium (Cr)

Chromium concentrations in the dissolved phase ranged from $0.44\mu\text{g/L}$ in the spring of 2023 at site 2 to the highest concentration of $1.84\mu\text{g/L}$ in the summer of 2023 at site 1.

In the particulate phase, the lowest concentration, $37.64\mu\text{g/g}$ dry weight, was recorded in the summer of 2023 at site 1, while the highest concentration, $410.92\mu\text{g/g}$ dry weight, was observed in the autumn of 2023 at site 2 (Table 1 & Fig. 4). The statistics revealed differences ($P < 0.05$) between seasons and studied sites in the cases of dissolved and particulate phase.

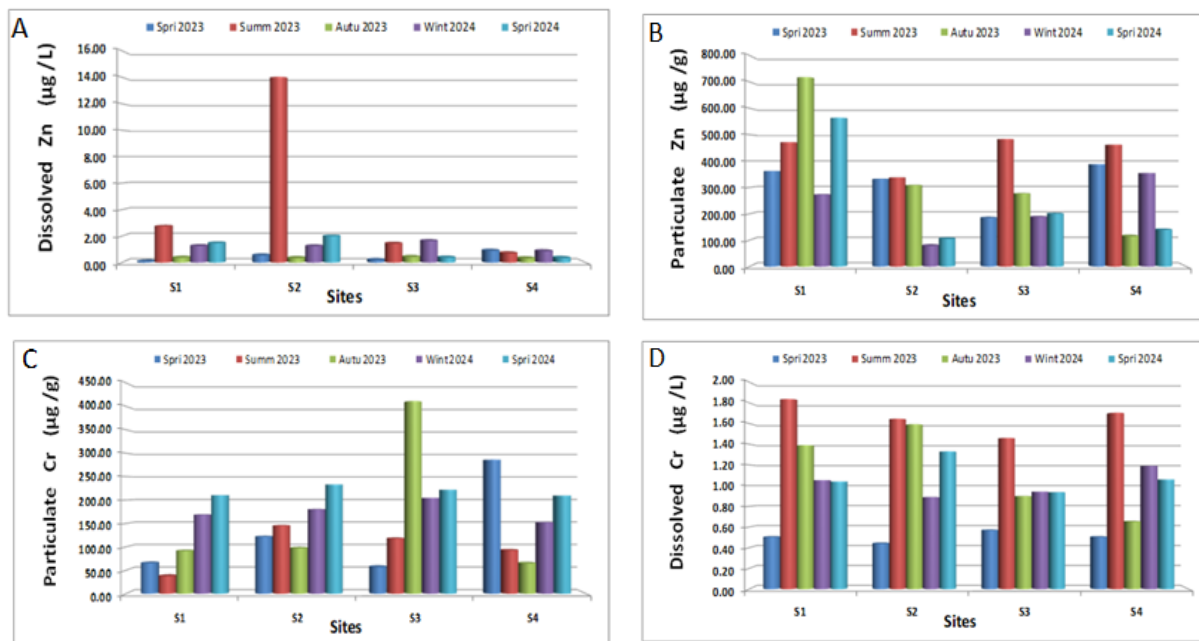


Fig. 4. Seasonal changes in the dissolved (A) and particulate (B) phases of zinc (Zn), and in the dissolved (C) and particulate (D) phases of chromium (Cr) in the water of the Al-Shamiyah River at the four sites during the study period

2. Heavy metals in sediments

1 – Copper (Cu)

Cu concentrations varied across the seasons. The lowest concentration in the exchangeable phase was observed at site 4 ($0.34\mu\text{g/g}$) in the winter of 2024, while the highest concentration, $5.05\mu\text{g/g}$, was recorded at site 2 in the spring of 2024.

In the residual phase, the lowest concentration, $8.5\mu\text{g/g}$ dry weight, was recorded at site 2 in spring 2024, and the highest concentration, $36.84\mu\text{g/g}$ dry weight, was observed at site 1 in autumn 2023 (Table 2 & Fig. 5). Statistical analysis revealed significant differences ($P < 0.05$) between seasons and study sites in both the exchangeable and residual phases.

2 – Manganese (Mn)

Manganese concentrations also showed seasonal variation. In the exchangeable phase, the lowest concentration was recorded at site 4 ($48.61\mu\text{g/g}$) in the spring of 2024, while the highest concentration, $313.61\mu\text{g/g}$ dry weight, was observed at site 2 in the same season.

For the residual phase, the lowest concentration, $288.68\mu\text{g/g}$ dry weight, was recorded at site 2 in the spring of 2024, and the highest concentration, $539.08\mu\text{g/g}$ dry

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weight, was observed at site 4 in the summer of 2024 (Table 2 & Fig. 5). Statistical analysis revealed significant differences ($P < 0.05$) between seasons and study sites for both the exchangeable and residual phases.

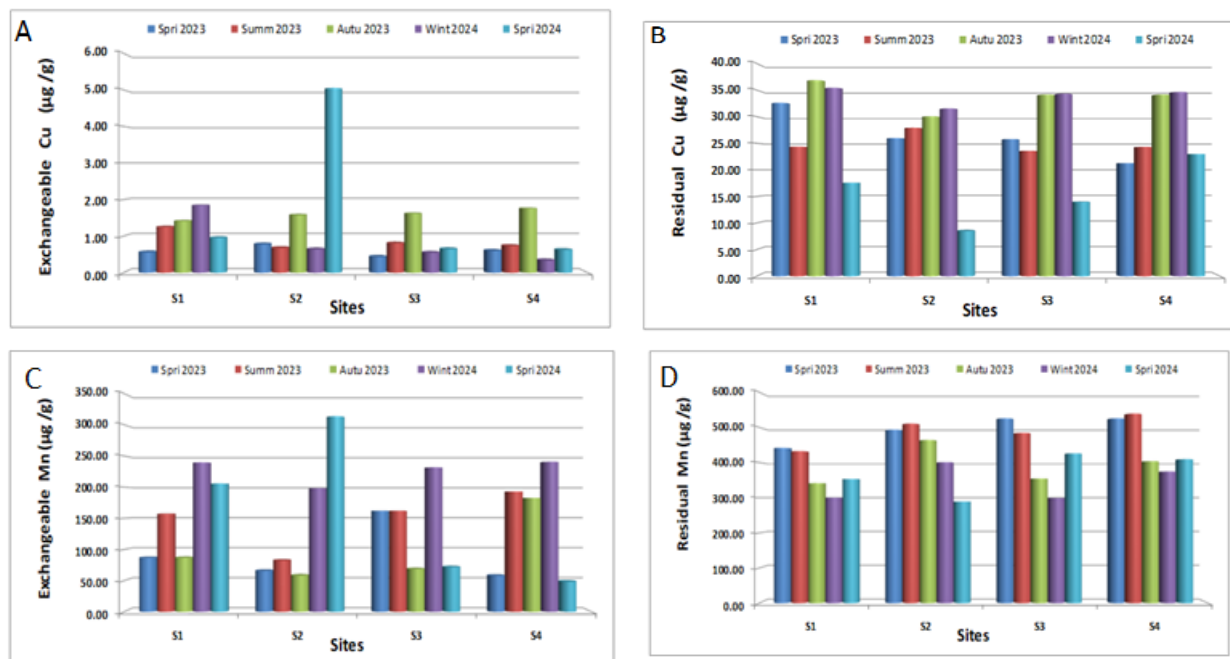


Fig. 5. Seasonal changes in the exchangeable (A) and residual (B) phases of copper (Cu), and in the exchangeable (C) and residual (D) phases of manganese (Mn) in the sediments of the Al-Shamiyah River at the four sites during the study period

3- Cadmium (Cd)

The lowest concentration of cadmium (Cd) in the exchangeable phase of sediments in the Al-Shamiyah River was $0.15\mu\text{g/g}$ at site 2 in the spring of 2023, while the highest concentration, $1.61\mu\text{g/g}$, was recorded at site 1 in the autumn of 2023.

In the residual phase, the lowest concentration of cadmium was $0.41\mu\text{g/g}$ at site 3 in the autumn of 2023, and the highest concentration, $3.04\mu\text{g/g}$, was observed at site 4 in the spring of 2024 (Table 2 & Fig. 6). Statistical analysis revealed significant differences ($P < 0.05$) between seasons and study sites for both the exchangeable and residual phases.

4 – Lead (Pb)

Lead concentrations varied across the seasons, with the lowest concentration in the Exchangeable phase recorded as $0.24\mu\text{g/g}$ at the first and second sites during autumn 2023. The highest concentration in this phase, $2.58\mu\text{g/g}$, was observed at site 4 during the summer of 2023.

In the residual phase, the lowest concentration of lead was $1.62\mu\text{g/g}$ dry weight at the third site in spring 2023, while the highest concentration reached $6.08\mu\text{g/g}$ dry

weight at the second site in spring 2024 (Table 2 & Fig. 6). Statistical analysis revealed significant differences ($P<0.05$) between seasons and study sites for both the Exchangeable and residual phases.

Table 2. Heavy metals concentration in sediments, exchangeable and residual phase ($\mu\text{g/g}$ dry weight) in Al-Shamiyah River

| Metals | | | Site 1 | | | | | Site 2 | | | | |
|---------|---------|-------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|
| samples | seasons | | Spring 2023 | Summer 2023 | Autumn 2023 | Winter 2024 | Spring 2024 | Spring 2023 | Summer 2023 | Autumn 2023 | Winter 2024 | Spring 2024 |
| Cu | Sedim. | Excha | 0.56 | 1.25 | 1.41 | 1.84 | 0.96 | 0.78 | 0.68 | 1.58 | 0.64 | 5.05 |
| | | Resid | 32.59 | 24.35 | 36.84 | 35.41 | 17.54 | 25.96 | 27.90 | 30.06 | 31.53 | 8.50 |
| Mn | Sedim. | Excha | 86.79 | 157.24 | 86.90 | 239.69 | 205.86 | 65.94 | 82.63 | 58.37 | 198.25 | 313.61 |
| | | Resid | 441.74 | 432.38 | 341.49 | 299.83 | 353.25 | 493.47 | 510.62 | 464.09 | 401.04 | 288.68 |
| Cd | Sedim. | Excha | 0.56 | 0.61 | 1.61 | 0.54 | 0.36 | 0.15 | 0.82 | 0.41 | 0.63 | 1.04 |
| | | Resid | 1.61 | 2.65 | 0.63 | 1.55 | 1.92 | 1.26 | 0.73 | 0.64 | 0.56 | 1.09 |
| Pb | Sedim. | Excha | 0.63 | 0.52 | 0.24 | 0.68 | 0.44 | 0.55 | 0.37 | 0.24 | 0.29 | 1.45 |
| | | Resid | 2.10 | 3.22 | 2.99 | 3.92 | 5.92 | 2.20 | 3.39 | 2.74 | 2.91 | 6.08 |
| Zn | Sedim. | Excha | 1.99 | 1.77 | 0.41 | 18.94 | 11.68 | 0.54 | 0.60 | 0.42 | 29.61 | 37.15 |
| | | Resid | 878.73 | 493.36 | 233.19 | 225.53 | 242.16 | 752.66 | 643.15 | 222.36 | 222.61 | 242.21 |
| Cr | Sedim. | Excha | 6.84 | 2.70 | 3.73 | 2.17 | 1.52 | 7.47 | 3.83 | 1.77 | 10.88 | 0.12 |
| | | Resid | 17.53 | 48.77 | 61.55 | 82.62 | 88.38 | 17.17 | 39.33 | 59.33 | 59.67 | 95.00 |
| | | | Site 3 | | | | | Site 4 | | | | |
| Cu | Sedim. | Excha | 0.44 | 0.81 | 1.62 | 0.55 | 0.65 | 0.60 | 0.74 | 1.76 | 0.34 | 0.63 |
| | | Resid | 25.77 | 23.56 | 34.14 | 34.30 | 13.94 | 21.26 | 24.31 | 34.14 | 34.63 | 22.99 |
| Mn | Sedim. | Excha | 161.95 | 161.95 | 68.69 | 231.73 | 71.89 | 58.02 | 193.11 | 182.72 | 240.65 | 48.61 |
| | | Resid | 525.64 | 484.41 | 354.72 | 299.05 | 426.12 | 525.64 | 539.08 | 403.78 | 374.59 | 409.53 |
| Cd | Sedim. | Excha | 1.51 | 0.59 | 0.47 | 0.64 | 0.54 | 0.75 | 1.53 | 0.49 | 0.56 | 0.34 |
| | | Resid | 1.51 | 1.58 | 0.41 | 1.47 | 1.45 | 1.70 | 1.31 | 0.71 | 1.32 | 3.04 |
| Pb | Sedim. | Excha | 0.44 | 2.49 | 0.33 | 0.51 | 0.42 | 0.69 | 2.58 | 0.32 | 0.42 | 0.48 |
| | | Resid | 1.62 | 3.46 | 1.79 | 3.53 | 5.33 | 5.08 | 3.15 | 2.26 | 3.59 | 5.47 |
| Zn | Sedim. | Excha | 0.81 | 0.81 | 0.48 | 11.77 | 8.16 | 0.43 | 1.92 | 0.78 | 6.77 | 2.67 |
| | | Resid | 906.39 | 690.66 | 222.34 | 235.46 | 243.94 | 778.19 | 529.78 | 219.13 | 227.01 | 235.76 |
| Cr | Sedim. | Excha | 6.47 | 2.25 | 2.36 | 1.33 | 0.72 | 7.21 | 2.78 | 1.84 | 1.51 | 0.48 |
| | | Resid | 19.16 | 46.31 | 67.88 | 79.17 | 85.65 | 27.32 | 45.20 | 77.79 | 83.79 | 70.81 |

*exchangeable phase = Excha. , residual phase = Resid.

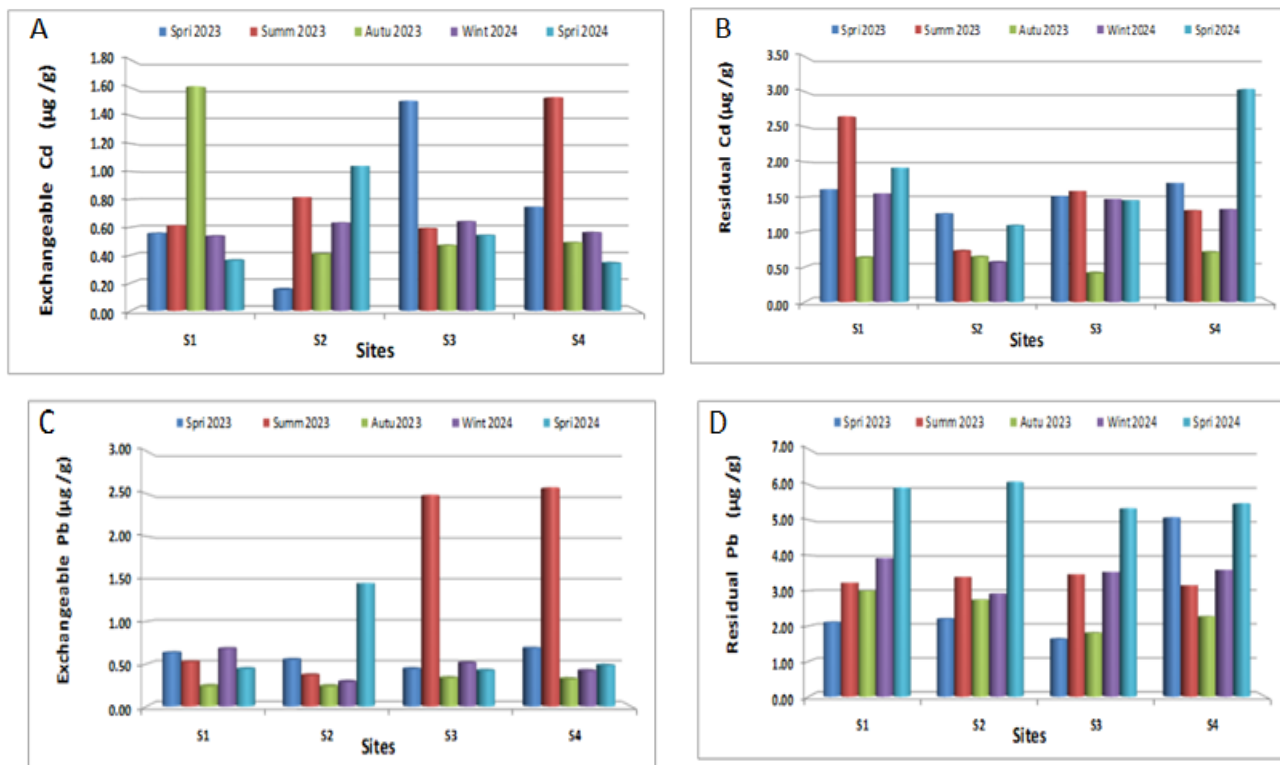


Fig. 6. Seasonal changes in the Exchangeable (A) and Residual (B) phases of cadmium (Cd), and the Exchangeable (C) and Residual (D) phases of lead (Pb) in sediments from the Al-Shamiyah River across the four sites during the study period

5 – Zinc (Zn)

The concentrations of this element also exhibited seasonal variation. The lowest concentration in the exchangeable phase of the sediment was recorded in autumn 2023, reaching $0.41\mu\text{g/g}$ at the first site, while the highest concentration was observed in spring 2023, at $37.15\mu\text{g/g}$ at the second site.

In the residual phase, the lowest concentration was recorded as $219.13\mu\text{g/g}$ dry weight in autumn 2023 at site 4, and the highest concentration reached $906.39\mu\text{g/g}$ dry weight in spring 2023 at site 3 (Table 2 & Fig. 7). Statistical analysis revealed significant differences ($P < 0.05$) between seasons and sites for both the exchangeable and residual phases.

6 - Chromium (Cr)

The lowest concentration of chromium in the exchangeable phase of the sediment during the year was $0.12\mu\text{g/g}$, recorded in the spring of 2024 at site 2. The highest concentration, $10.88\mu\text{g/g}$, was observed at the same site in the winter of 2024.

For the Residual phase, the lowest concentration was $17.17\mu\text{g/g}$, recorded in the spring of 2023 at site 2, while the highest concentration reached $95.00\mu\text{g/g}$ at the same site in the spring of 2024 (Table 2 & Fig. 7).

Statistical analysis indicated significant differences ($P < 0.05$) between seasons and sites for both the Exchangeable and Residual phases.

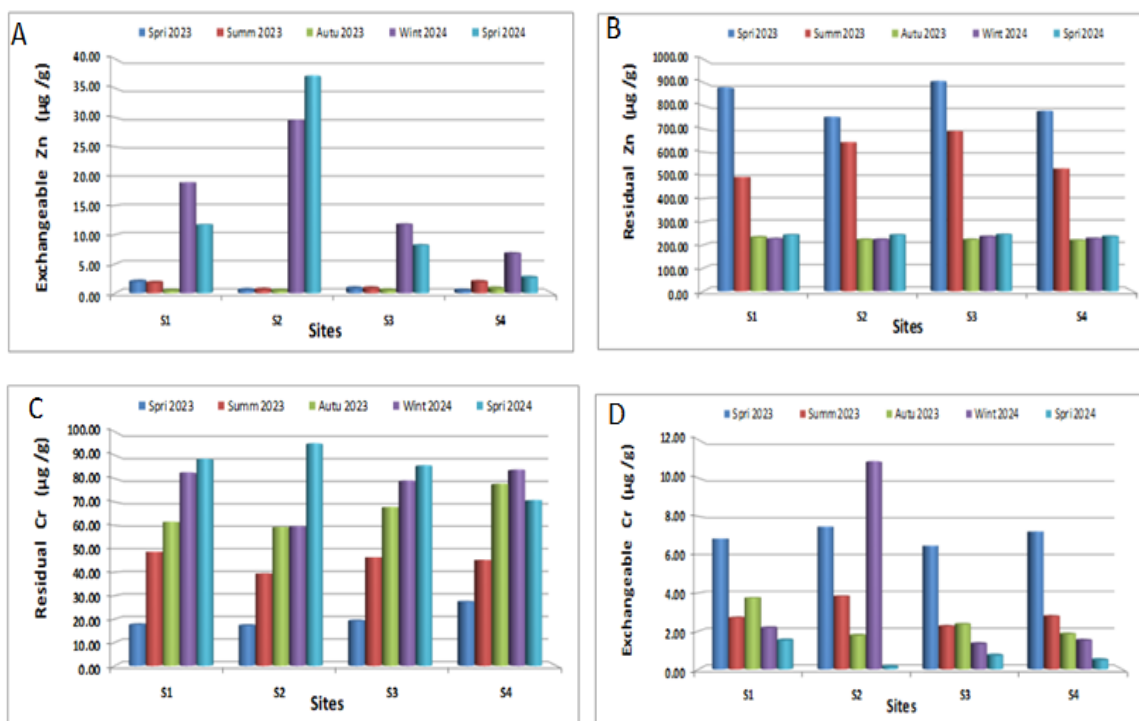


Fig. 7. Seasonal changes in the Exchangeable and Residual phases of (Zn) **A** and **B**, respectively, and Chromium (Cr) concentrations **C** and **D**, respectively, in the sediments of the Al-Shamiyah River at four sites during the study period

4. Discussion

4.1. Heavy elements in water

The increase in heavy metals in the waters of the Al-Shamiyah River is primarily due to soil erosion contaminated with these metals, sewage waste discharge, and agricultural activities. Agricultural and human activities are major contributors to the accumulation of heavy metals in aquatic ecosystems. Due to their high stability, these metals can accumulate in elevated concentrations in river water (Ali *et al.*, 2019; Chiudioni *et al.*, 2024).

The rise in heavy metals in the Al-Shamiyah River is also a result of waste discharge from households and agricultural runoff into the river. These discharges vary by site and season. Additionally, the use of fertilizers on lands adjacent to the river and the drainage of water from surrounding areas contribute to the contamination. As the river often receives water without prior treatment, this further increases the concentration of dissolved heavy metals (Sekabira *et al.*, 2010). This finding aligns with the study by Audry *et al.* (2004) on the Lot River in France and with the research of Al-Khafaji *et al.* (2011) on the Euphrates River near Al-Nassiriya, Iraq. Furthermore, sewage water, which

contains high levels of heavy metals, flows into the Al-Shamiyah River, as the river serves as a drainage outlet for this waste. Unfortunately, this water is contaminated with agricultural residues, household waste, and other anthropogenic pollutants, all of which carry varying amounts of heavy metals, leading to increased concentrations in the river (**Mojiri & Aziz, 2011**).

This observation is consistent with the study of **Al-Ghanimy and Al-Rekabi (2023)** on the Euphrates River in Iraq. The decrease in the concentration of heavy metals in the dissolved phase, compared to the particulate phase, in the Al-Shamiyah River during the current study is also in agreement with the findings of **Hassan *et al.* (2010)** addressing the Euphrates River. The reduction in the concentration of elements in the dissolved phase may be attributed to algae taking up and concentrating these elements in their bodies during their growth and reproduction cycles (**Ankit & Korstad, 2022**).

The higher concentrations of heavy metals in the summer months may be linked to higher temperatures and increased evaporation rates, which concentrate the metals in the water (**Rasheed *et al.*, 2024**). Conversely, lower concentrations, or even an absence of these metals, can be attributed to increased river water levels due to environmental factors such as rainfall and flooding, which dilute the concentrations of heavy metals (**Trakulsrichai *et al.*, 2017; Younis & Saeed, 2023**).

The concentration of the studied elements also varied within a single site and season. For instance, the increase in cadmium concentrations during certain seasons may be related to the use of phosphate fertilizers, which contain high levels of cadmium, as well as heavy water discharges into the river (**Al-Bassam, 2011**).

The high concentration of chromium in the Al-Shamiyah River can be attributed to its increased use in electroplating, cooling tower drainage, and the ceramic industry. Industrial processes, including cotton and wool dyeing, metal manufacturing, and leather tanning, produce large quantities of wastewater and sediments containing heavy metals, including chromium, thus raising its concentration in river water (**Mittal *et al.*, 2021**). Additionally, hexavalent chromium (Cr(VI)) is more stable than trivalent chromium (Cr(III)), and in the presence of manganese oxides in river water, Cr(III) can be converted into the more stable Cr(VI), contributing to the increased chromium concentrations (**Bourotte *et al.*, 2009**). The elevated levels of manganese in the water may be due to its increased mobility under basic conditions. While manganese is essential for organisms' metabolic processes at low concentrations (**Peralta Vaidya *et al.*, 2009**), its concentration can become harmful to living organisms once it exceeds a certain threshold (**Ali *et al.*, 2013; Geist & Hawkins, 2016**).

The rise in zinc concentrations in the Al-Shamiyah River may also be linked to the discharge of residential and agricultural wastewater into the river. Agricultural runoff, especially from lands near the river, may contain pesticides such as zinc phosphide, used as a rodenticide, as well as other pollutants. Drainage from these areas can further

contribute to the increased zinc concentration in the river (**Al-Maliky, 2005; Trakulsrichai *et al.*, 2017**).

The concentration of lead and cadmium in the Al-Shamiyah River is lower than that in the Euphrates River, as reported by **Karem *et al.* (2024)**. Similarly, the concentrations of heavy metals (Pb, Cd, Zn) in the Al-Shamiyah River are lower than those found in the Tigris River, as documented by **Ahmed and Al-Shandah (2024)**. However, the concentration of heavy metals in the Al-Shamiyah River is higher than the levels reported by **Al-Taher *et al.* (2020)** in their study of the Euphrates River. Both studies observed seasonal and spatial variations in the concentration of heavy metals in the waters of the Euphrates and Levant rivers. Moreover, the current study found significant seasonal and spatial differences in the concentration of heavy metals in the Al-Shamiyah River, with certain sites and seasons showing higher or lower levels of contamination. These findings are consistent with the study of] **Rasheed *et al.* (2024)** on the Tigris River.

Heavy metals that enter aquatic ecosystems do not remain in dissolved form for a long time but rather tend to bind to suspended particles such as clay, silt, and aquatic organisms, accumulate in aquatic plants or other organisms, and descend to the bottom to bind with the organic materials found in the sediments (**Edward *et al.*, 2013**).

Some factors help heavy elements bind to suspended particles in the water column, including turbidity, pH, suspended solids, and carbon dioxide. pH plays a role in transforming heavy elements from dissolved to particulate and vice versa. Suspended solids also help heavy particles settle on a river's bottom. When bound to it, there is also the presence of organic materials in the sediments, which, upon decomposition, produce carbon dioxide, which reacts with water and produces carbonic acid, acting to lower the pH; this in turn increases the dissolution and the release of heavy elements from the sediments and vice versa (**Ideriah *et al.*, 2012**).

This study revealed that heavy metals, particularly manganese (Mn), chromium (Cr), zinc (Zn), and copper (Cu), are present at high concentrations in the particulate phase of the Al-Shamiyah River. This can be attributed to rainfall, which leads to soil erosion and runoff, as well as the drainage of agricultural lands into the river after irrigation during the agricultural season (**Kar *et al.*, 2008**).

The study also demonstrated seasonal variations in the concentration of heavy metals in the particulate phase of the river, likely due to changes in water properties, including organic and inorganic compounds, as well as other pollutants. Dilution factors from water flow, along with the activities of aquatic organisms (such as microorganisms, algae, and plants), may also contribute. These organisms can absorb significant amounts of elements from the surrounding environment (**Salman, 2011**).

The rise in heavy metal concentrations in the particulate phase of the Al-Shamiyah River may be due to the tendency of many heavy metals to adsorb onto suspended particulate matter in the water. When water samples are collected and filtered, the concentration of heavy metals in the suspended phase is generally higher than in the

dissolved phase (**Al-Ghanimy & Al-Rekabi, 2023**). Additionally, the relatively low concentration of heavy metals in the particulate phase could be because particles containing these elements tend to precipitate under basic conditions, as the Al-Shamiyah River is alkaline (**Weiner, 2000**).

The concentrations of manganese, chromium, copper, lead, and zinc in the particulate phase of the Al-Shamiyah River are higher compared to those found in the Euphrates River in the study by **Salman *et al.* (2015)**, with the exception of cadmium and lead, which were less concentrated in the Al-Shamiyah River. This difference may be attributed to the Al-Shamiyah River's exposure to both agricultural and urban pollutants, given the large number of areas it passes through (**Callender & Rice, 2000**).

Undoubtedly, environmental pollution with heavy metals in the Al-Shamiyah River is worsening, likely due to anthropogenic activities that have increased the presence of heavy elements in both the water and sediments (**Aljanabi *et al.*, 2022**). Heavy metal pollution may also result from industrial processes, pesticide use, and fertilizer runoff from agricultural lands surrounding the river (**Schuler & Relyea, 2018; Mathur & Sherry, 2023**).

The growing population in the study area may further impact the aquatic ecosystem of the river, as increased human needs drive industrial and agricultural activities that release heavy metals into the water (**Mishra *et al.*, 2022**). The distribution of heavy metals between dissolved and particulate phases, as well as their deposition in sediments, is influenced by processes such as absorption, co-sedimentation, and hydrolysis. These processes cause the concentration of metals in the Al-Shamiyah River to vary between the water and sediment phases, as well as across the alternating and residual phases (**Varol & Sen, 2012**).

For example, industrial activities contribute to the release of heavy metal ions, which are transported and deposited in the aquatic ecosystem (**Mishra *et al.*, 2022**). **Al-Robai (2013)** noted that, in the Hilla River, heavy metals are concentrated in the suspended phase and less in the dissolved phase.

Furthermore, the concentrations of heavy metals in the dissolved phase of the Al-Shamiyah River during this study exceeded the standard limits set by the **WHO (2006)** for most elements, except for zinc, which did not exceed the limits. However, cadmium and chromium concentrations exceeded the limits established by the **CCME (2007)**.

4.2. Heavy elements in exchanged and residual phase in sediments

Sediments at the bottom of rivers act as a repository for heavy metals and other pollutants that enter the water column. These contaminants settle when they bind to particles such as clay, silt, and sand, eventually sinking to the riverbed. The sedimentation process is influenced by various factors, including water movement, seasonal changes in thermal distribution, the activity of benthic aquatic organisms, and fluctuations in water levels during the agricultural season (**Cai *et al.*, 2018**).

Pollutant concentrations in sediments, including heavy metals, are often higher than in the dissolved or fine particulate phases. This is because many heavy metals in the water column are associated with plankton. When these particles settle to the riverbed, they contribute to higher metal concentrations in the sediments (**Aprile & Bouvy, 2010**).

This study showed that concentrations of heavy metals extracted from sediments in the alternating and residual phases varied between seasons and sites. These variations are likely due to the release of high concentrations of these elements into the river from human sources, including wastewater containing organic materials that form complexes with the metals, which are then deposited in the sediments (**Salman *et al.*, 2015**).

The study also found that the concentration of heavy metals in the residual phase was higher than in the alternating phase of the sediment. This finding is consistent with other studies that reported high concentrations of metals such as zinc, manganese, chromium, and lead in the sediments of the Al-Shamiyah River, as well as in the Euphrates and Diyala Rivers (**Saber *et al.*, 2023; Mijbas *et al.*, 2024**).

The variation in heavy metal concentrations in the sediments of the Al-Shamiyah River may be attributed to differences in the alternating and residual phases between sites and across seasons. Factors such as sediment type at each site, the distance the river travels, and human activities all influence these variations. Pollutant sources differ by site and season. For instance, urban pollution typically increases copper concentrations, agricultural runoff raises zinc levels, and industrial pollution tends to elevate chromium concentrations in the river (**Haghnazar *et al.*, 2021; Ustaoglu, 2021; Sojka & Jaskula, 2022**).

Changes in pH can also influence the concentration of heavy metals in sediments. As pH becomes more acidic, more metals dissolve from the sediments into the water column, which reduces their concentration in the sediment phase. Variations in pH, temperature, dissolved oxygen, and surface water flow rates all contribute to fluctuations in heavy metal concentrations (**Zaimee *et al.*, 2021**). The high concentration of heavy metals in the sediments of the Al-Shamiyah River is likely due to the sediments acting as the final sink for pollutants from both natural and anthropogenic activities that enter the river and carry high levels of heavy metals (**Saeed & Shaker, 2008**).

Fertilizers and pesticides used on agricultural lands near the river contribute to increased organic material in the water. The surface area of this material helps adsorb heavy metals, which are then deposited at the river's bottom, raising the concentration of metals in the sediments (**Turki, 2007**).

The high concentration of heavy metals in the sediments may also result from the decomposition of phytoplankton and other organisms, which accumulate and deposit on the riverbed. Additionally, the release of sewage water, household waste, and runoff from lands adjacent to the river, as well as atmospheric deposition, contributes to elevated metal concentrations in the sediments of the Al-Shamiyah River (**Nakanishi *et al.*, 2004**).

The increased concentration of heavy metals in the alternating and residual phases in the sediments of the Al-Shamiyah River may also be due to the formation of carbonates, oxides, hydroxides, and silicates with these elements, which causes them to precipitate due to the high pH of the river (Agarwal, 2009). Furthermore, the nature and texture of the sediments influence the concentration of metals. As the concentration of metals increases, the sediment size tends to decrease, with sandy sediments containing lower concentrations of heavy elements (Al-Juboury, 2009).

Sediments in rivers, including those in the Al-Shamiyah River, can be polluted by heavy metals such as cadmium and lead, which are toxic to organisms. These metals can be transmitted to animals that drink contaminated water and to plants irrigated with polluted water. Water contaminated with heavy metals is often used for domestic and agricultural purposes (Kubier *et al.*, 2019; Okereafor *et al.*, 2020; Vajargah, 2021).

The current study shows that the concentration of heavy metals in the Al-Shamiyah River is high, but fluctuates over time. The entry of heavy metals into the river varies due to the amount of agricultural and domestic wastewater containing heavy metals that enters the river at the study sites. High concentrations of heavy metals may also result from sewage discharge into the Al-Shamiyah River. Furthermore, high temperatures play an important role in the dissolution of metals in water. Al-Taher *et al.* (2020) observed this phenomenon in the Euphrates River, where increased warming and bacterial decomposition, along with other environmental factors, lead to higher metal concentrations in the water (Hu *et al.*, 2022; Yu *et al.*, 2022).

The results of this study indicate that the concentrations of heavy metals in the sediments of the Al-Shamiyah River did not exceed the standard limits set by the CCME (2001) and CBSQG (2003).

CONCLUSION

The study revealed the following concentrations of heavy metals in the water of the Al-Shamiyah River:

- Dissolved phase: $Mn > Zn \geq Cr > Cu > Cd > Pb$
- Particulate phase: $Zn > Cr > Mn > Cu > Cd > Pb$

In the sediments of the Al-Shamiyah River, heavy metal concentrations were observed in the following order:

- Exchangeable phase: $Mn > Zn > Cr > Cu > Pb > Cd$
- Residual phase: $Zn > Mn > Cr > Cu > Pb > Cd$

Manganese (Mn) was found to be the highest in both the dissolved phase in water and the exchangeable phase in sediments, while zinc (Zn) had the highest concentration in the particulate phase of water and the residual phase of sediments.

Pollution with heavy metals in aquatic ecosystems is a significant environmental issue due to its harmful effects on human health, prompting researchers to investigate the underlying causes. To mitigate the risks, it is essential to treat heavy metal-contaminated water and sediment. However, conventional methods often have limitations, particularly

regarding their environmental impact. To address these challenges, we suggest exploring biological remediation techniques and integrating various approaches for effectively removing heavy metals from aquatic ecosystems.

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