

Detection of Some Heavy Metals Used in the Surface Soil (Sediments) from Khirbet Al-Samra in Jordan

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ABSTRACT

Spatial analysis was used to analyze the environmental quality of soil in the Al-Zarqa region in order to identify sources and estimate heavy metal concentrations, which aided in the assessment of soil quality and heavy metal pollution. The primary goal of this study was to assess the environmental impact of heavy metal pollutants in the Al-Zarqa region. To assess pollution levels, the concentrations of Cu, Mn, Cd, and Pb were measured in surface soil (sediment) samples collected from Khirbet al-Samra. A total of sixteen samples were analyzed. The elevated levels of Cu and Cd are primarily attributed to various sources such as the weathering of nearby rock formations and the release of agricultural waste materials. An evaluation of sediment contamination was conducted using pollution indicators including Geo-accumulation index (I_{geo}), enrichment factor (EF), and pollution load index (PLI). Spatial distribution analysis was used to determine the distribution pattern of each metal. The results revealed that metal concentrations (Cu, Pb, and Mn) are higher, while Cd concentrations are lower than the maximum allowed limits. The results from the EF analysis indicated elevated concentrations of Cu and Cd in the sampled area. In terms of the Igeo analysis of Khirbet al-Samra sediments, it was found that the concentrations of Pb, Cu, and Mn are within safe levels and relatively unaffected by human activities, whereas the concentrations of Cd exceed the mean values, suggesting a higher level of contamination specifically for cadmium. The potential sources of heavy metals in the investigated area were identified using factor analysis, and the geographical distribution of heavy metals was shown using spatial distribution. The examination of correlation coefficients revealed diverse relationships between the different parameters, depending on the source of input for each metal.

Keywords: heavy metals, surface soil, sediments, detection, contamination, pollution, trace elements, Khirbet al-Samra.

INTRODUCTION

In contemporary societies, environmental pollution poses a significant challenge. Among the various forms of pollution, the high contamination of aquatic environments with heavy metals is particularly concerning due to their non-biodegradable nature. Elevated levels of heavy metals could potentially influence food quality and safety [1]. These metals primarily enter marine environments through natural processes such as rock erosion and weathering, as well as through human activities including industrial operations,

sewage disposal, runoff from land, and agricultural practices [2]. Heavy metals introduced into aquatic environments undergo key processes such as adsorption, flocculation, and co-precipitation, becoming immobilized in stream sediments. Sediments play a crucial role in retaining or releasing metals within marine ecosystems through various recycling mechanisms that affect the water column [3]. Different processes lead to the interaction of heavy metals with solid phases, including adsorption onto hydrous magnetic oxides and ferric compounds, association with clays or natural organic substances, absorption by fine-grained

inorganic particles, and direct precipitation as new solid phases [4]. Numerous experiments have demonstrated that sediment metal concentrations serve as sensitive indicators of contamination in aquatic environments [5–8]. Various tools and indices such as the EF, PLI, and I_{geo} are utilized to assess sediment contamination and understand the contributions of both human activities and natural sources in river systems [9]. Heavy metals are generated through physical and chemical weathering of rocks into the air and sediment. Other sources include atmospheric deposition, forest fires, oceanic spray, wind erosion, plant exudates, and volcanic activity [10]. Fossil fuel combustion is also a significant contributor, releasing metals like Pb, Cd, As, Se, Sb, Ba, Cu, Mn, Zn, and V into the environment, which can contaminate soil and water during combustion or through ash disposal [11]. Several studies have investigated heavy metal distributions and contamination levels in various environments. For instance, a study in Jordan focused on heavy metal distribution in Khirbet al-Samra sediment, highlighting anthropogenic sources of contamination [12]. Another study assessed contamination in Wadi al-Arab Dam sediments, providing insights and metrics for measuring contaminants and aiding environmental policymaking [4]. Multivariate analyses, including correlation matrix and factor analysis, were employed to better understand pollutant dynamics in these studies. Furthermore, remote sensing data and analytical techniques have been used to evaluate heavy metal contamination in areas like Wadi Shu'ayb soil sediment, revealing complex geochemical interactions and indicating potential environmental risks [14]. Similarly, an analysis of sediment samples from Wadi Al-Arab in Jordan revealed safe levels of certain metals but raised concerns about pollution and high concentrations of Zn and Cd [15]. Several techniques have been used in the literature to determine chemical element concentrations in sediments, water, and fish. These techniques include X-ray fluorescence (XRF) [16], inductively coupled plasma atomic spectroscopy (ICP-AES) [17], and ICP-MS [18]. Monitoring studies of trace element concentrations in Khirbet al-Samra sediments have become critical for determining the contamination effects of various pollutant sources. The study also investigated analyzing heavy metal concentrations in sediments from Khirbet al-Samra in Jordan using Igeo, EF and PLI.

MATERIALS AND METHODS

Sampling and sample preparation

Ten stream sediment samples were taken during the current year 2024 (January, February, March, April, and May) from the main Khirbet al-Samra in the study area, which is in the center of Jordan. The samples were kept in a deep freezer at $-20\text{ }^{\circ}\text{C}$ to preserve them until the freeze-drying procedure was performed. In the end, the samples were freeze-dried for 48 hours in an Edwards Modulyo Freeze Dryer at $-40\text{ }^{\circ}\text{C}$ and 100 mTorr pressure. This receive wastewater from highly populated areas of Amman the capital and Zarqa governorate. This wastewater is the largest wastewater treatment plants in Jordan, and uses advanced technologies to remove organic matter and other impurities from the wastewater. Multiple processes are carried out in the plant, that include preliminary screening, aeration, sedimentation, coagulation, filtration and disinfection, with the aim of removing harmful substances and achieving the required quality standards [19]. The depth of the samples was 0 to 25 cm. The samples were taken in order to examine all of the sediment in the study area. Then debris and large stones were removed. Following air drying to remove very large particulates and obtain a homogenous sample, sediment samples are put onto a stainless steel sieve. Samples are then transferred to marked, pre-washed, polyethylene bags that are easily transported to the lab for additional sample processing and examination. Atomic Absorption Spectrophotometer measurements were made to determine the metal concentrations in the samples of digested sediment.

Chemical and reagents

All reagents used in the experiments were of analytical grade purity. Milli-Q water with a resistance of 18.8 MS_2 was utilized. Sodium acetate, nitric acid (with 69% purity), and acetone were procured from GCC, UK. Potassium dichromate, o-phenanthroline indicator, and H_3PO_4 were obtained from Fluka, Switzerland. Ferrous ammonium sulfate was sourced from BDH, England. Isopropyl alcohol from Fisher Scientific, USA, and ammonium acetate from Scharlau Chemie S.A, Spain, were also employed. Additionally, n-hexane from Frutarom, UK, and a PAHs reference standard solution were used in the experiments. To

achieve a great preconcentration factor, the solvent was evaporated under a gentle nitrogen steam and reconstituted with 1 mL of Milli-Q water with 100 g/L of internal standards. Before analysis, the extracts were filtered using Chromafil Xtra PET-20/25 syringe filters with a pore size of 0.20 μm from Machery-Nagel (Düren, Germany).

Sample extraction

The extraction method employed in contamination studies, known as the ammonium acetate-EDTA extraction, was utilized to assess the heavy metal content with improved accuracy. This extraction procedure is commonly used to evaluate both main and trace elements such as Zn, Fe, Cr, Pb, Ni, Cd, Cu, Mo, Co and Mn. The extraction solution was prepared by combining 0.5 M NH_4Ac (ammonium acetate), HAc (acetic acid), and 0.02 M EDTA at a pH of 4.65. The procedure involved dissolving 38.5 grams of NH_4Ac in 500 ml of water along with 25 ml of acetic acid, then adding 5.845 grams of EDTA, and finally adjusting the volume to one liter with deionized water. Subsequently, 20 grams of air-dry soil samples were placed in a 300 ml Erlenmeyer flask, and 100 ml of the prepared extracting solution was added. The flask was then mechanically shaken for 30 minutes to facilitate the extraction process. After shaking, the suspension was filtered, and the filtrate was collected in a polyethylene flask for further analysis [20].

Sample preparation

The sample consisted of seventeen sediment samples, which were then dried for 2 hours in an oven set at 55 °C. Specifically, particles smaller than 63 μm (0.05 mm) in size were selected for analysis, as this size is considered optimal for analytical purposes in arid and semi-arid regions [21]. The geological sample, measured at approximately 2.5 grams, was treated with 100 ml of 1N HCl in a beaker. The mixture was allowed to react for about 20 minutes while being stirred with a glass rod. After 20 minutes, the solution was filtered, and the clear filtrate was collected in a new beaker. For the titration process, 20 ml of the sample solution was transferred to a conical flask. NaOH from a burette was then titrated into the flask, with 4 or 5 drops of bromophenol blue indicator added. The titration continued until the color of the sample solution in the flask turned blue, indicating the end-point of the reaction. The volume of NaOH

used during titration and the reading on the burette were noted. This process of titration was repeated three times, and the average value was calculated as the titration value for the sample [22]:

$$\text{Carbonate percent} = 10 \times (\text{Blank value} - \text{Test value}) \quad (1)$$

Determination of organic matter percent by ashing method

Using the linear relationships among LOI values and the contents of organic and inorganic carbon, sequential loss on ignition (LOI) is an easy approach to evaluate the quality of sediment organic matter and carbonate minerals [22]. At 100 °C, a mass of sediment can be dried and then heated at 550 °C for 1 hour. Combustion at 550 °C converts organic matter (OM) to CO_2 according to the reaction; $\text{CH}_2\text{O} + \text{O}_2 \rightarrow \text{CO}_2 + \text{H}_2\text{O}$.

The percent of (OM) calculated by the following Equation [23]:

$$\% \text{ of (OM)} = \frac{WB - WA}{WB} \times 100 \quad (2)$$

where: WB – weight sample before ashing, WA – weight sample after ashing.

RESULTS AND DISCUSSIONS

Enrichment factor

Enrichment factor (EF) analysis was first presented to evaluate trace element concentration mathematically as [24]:

$$EF = (M/Fe)_{\text{sample}} / (M/Fe)_{\text{background}} \quad (3)$$

Values of EF in the study area are shown in Figure 1. The EF results revealed high concentrations of Cd and Zn in the analyzed samples, as depicted in Figure 1. This indicates a potential risk of pollution due to the elevated levels of Zn and Cd, especially considering that an EF value exceeding 40 is indicative of extreme pollution [14]. These heavy metals are likely derived from anthropogenic sources, such as pesticides and fertilizers used in agricultural activities, which contribute to environmental contamination. It's important to note that variations in the contribution level of each metal to sediments, or differences in the rate of extraction of each metal from the sediments, can lead to fluctuations in EF values [25]. This variability highlights the complex dynamics involved in heavy metal pollution and

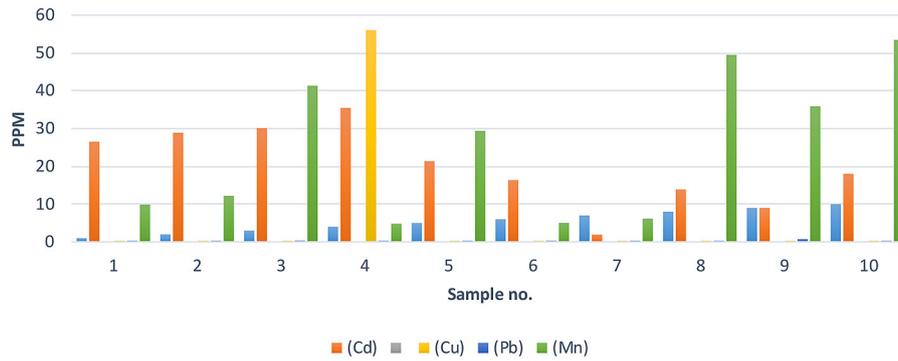


Figure 1. Enrichment factor of the heavy metal in the studied 10 samples

underscores the need for comprehensive assessments and monitoring strategies to address environmental risks effectively.

Geoaccumulation index (I_{geo})

Geoaccumulation index (I_{geo}), announced to determine the extent of metal accumulation in sediments [26]. I_{geo} is mathematically expressed as:

$$I_{geo} = \log_2 (Cn/1.5Bn) \quad (4)$$

The I_{geo} values of the studied samples are listed in Figure 2. The I_{geo} distribution in the study samples is depicted in Figure 2, showing varying percentages of I_{geo} depending on the type of metal and its location. Specifically, Fe, Pb, and Cu consistently show I_{geo} grades below zero (indicating unpolluted conditions) across all stations, suggesting that sediments in the sample region have natural background levels for these metals. On the other hand, Mn, Zn, and Cd exhibit I_{geo} grades ranging from moderately to strongly polluted, with variations based on the sample location

and concentrations of these elements. Notably, Zn shows the highest I_{geo} grades, exceeding five in some areas, indicating extreme pollution in sediment samples. This highlights that the sediments in Khirbet al-Samra are experiencing background concentrations for Zn, Mn and Cd, with limited changes due to anthropogenic influences. The sources of these hazardous metals can be traced back to industrial waste and additives used in gasoline by industries and automobiles, contributing to environmental pollution in the region [27]. Understanding these distribution patterns and the sources of pollution is crucial for developing effective strategies to mitigate environmental contamination and safeguard ecosystems.

In addition, they might be derived from corrosion agricultural activities in the Khirbet al-Samra. Cu is known to have low limits of detection in NAA of geological materials due to the brief half-life of ^{66}Cu of 5.05 minutes and the need to wait 15–20 minutes for the ^{28}Al half-life of 2.24 minutes. As evidenced by the results from the reference materials, the uncertainty for copper

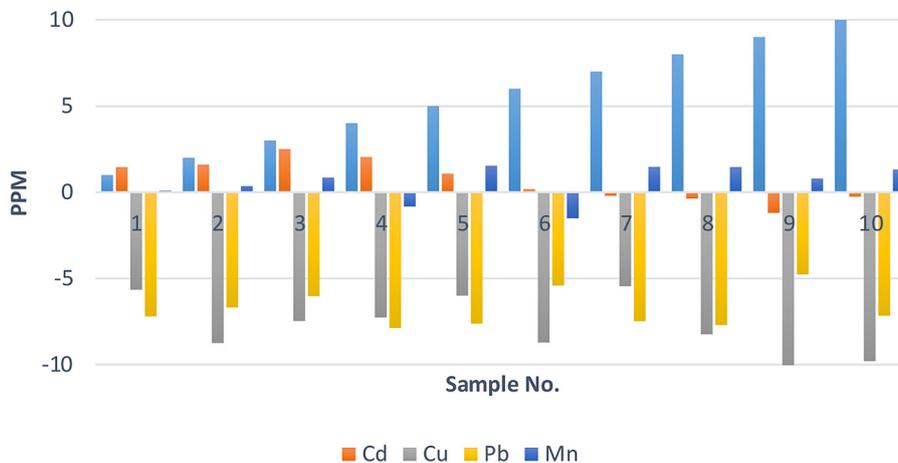


Figure 2. The I_{geo} values of the heavy metal for study samples

is extremely large or below detection limits. As a result, copper concentrations were not included in the sediments. Copper produced higher detection limits for fish, and the outcomes are presented. Iodine can be vaporized in samples based on the chemical bonds present. Thus the findings for iodine should be interpreted as a lower limit [27].

Pollution load index (PLI)

This analysis has measured the following pollution load index (PLI) expressed as [28]:

$$PLI = (CF1 \times CF2 \times CF3 \times \dots \times CFn)^{1/n} \quad (5)$$

The PLI values of study area listed in Figure 3. The severity and variation in pollution levels within the study area were assessed using the PLI method. This index serves as a guide for comparing the degree of pollution across different locations [29]. The results of the study show that the CF values for metals such as Fe, Cu, and Pb are low, all being less than 1. This indicates relatively low levels of pollution for these particular metals in the sediment samples analyzed. The fluctuations observed in the indices are attributed to the varying sensitivity of different indices towards sediment pollutants [30]. Each index used in the assessment may have different thresholds and criteria for evaluating pollution levels, leading to fluctuations in the calculated indices. Understanding these variations and sensitivities is essential for accurately assessing pollution severity and developing targeted mitigation strategies to address environmental challenges in the study area.

PLI results indicate that there are no pollution threats in the study area as low indices reported. Figure 3 depicts the differences in mean

concentrations among all four sites. PLI values (CF) results show that all sites' concentration means are significantly different from one another. However, concentration values in site four differ significantly from those in the other three sites, most likely due to its proximity to the Khirbet Al Samra wastewater treatment plant (the largest in the country). The element obtained was Zn, with a typical varying concentration in Jordanian environments of 121 to 158 mg/kg. However, the amount of Zn in the Zarka River sediments measured in this study ranged between 17.2 and 106.7 mg/kg, which is less than the average range [30]. In a return to previous literature, Igeo values show different levels of contamination for different metals for a number of reasons [34–36].

Sediment contamination assessment (statistical analysis)

The strength of the relationship between variables is indicated by the correlation coefficient, with values near 1 or -1 indicating a strong relationship. Conversely, low values closer to zero suggest a weak or no relationship between variables. Generally, parameters with a correlation coefficient (r) greater than 0.7 can be considered very similar to each other, while values between 0.5 and 0.7 indicate a mild correlation [31]. The correlation matrix, as shown in Figure 4, displays the correlation between the calculated elements. By examining the values in the correlation matrix, one can identify which variables have strong positive correlations (values close to 1), strong negative correlations (values close to -1), or weak correlations (values closer to 0). This information is crucial for understanding the interrelationships between

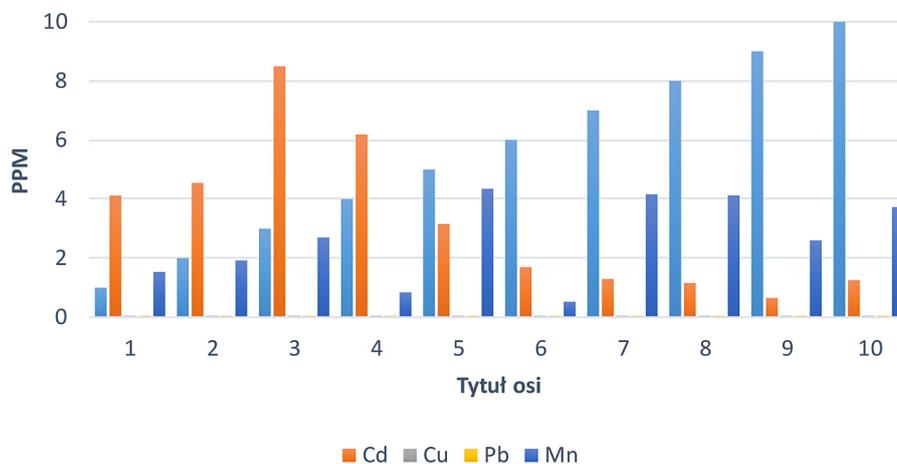


Figure 3. PLI values (CF) of the heavy metal for the studied samples

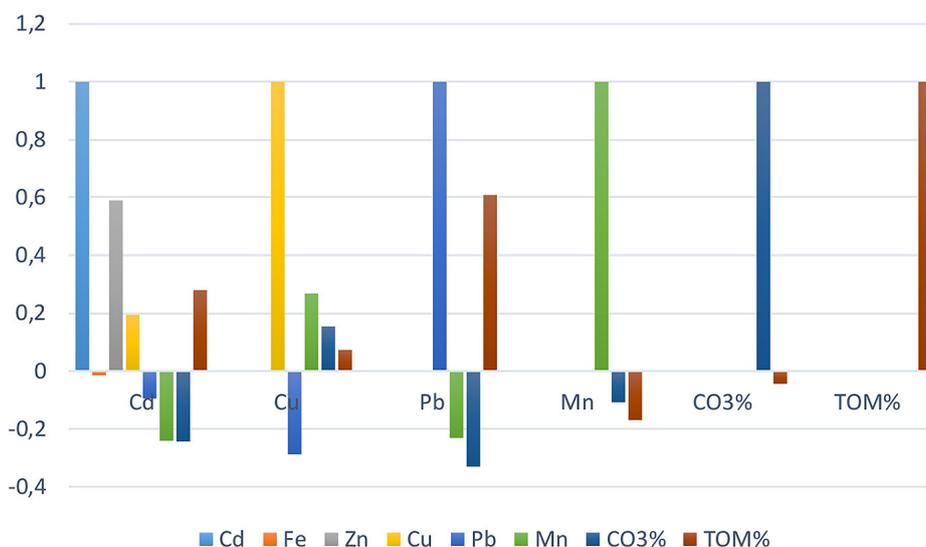


Figure 4. Correlation matrix of the heavy metals for the studied samples

different parameters and their potential impacts on the study area or system under investigation.

The reason for the increase in heavy metal pollution in that region is increased urbanization, industrialization, traffic, oil refinery emissions, and a mix of anthropogenic sources. Three parameters are used to define the pollution level in the sediment: EF, PLI, and I_{geo} [25]. Strontium (Sr) had the highest concentration of any metal studied in sediment samples. The results revealed a variation in elemental concentration between sites that is associated with the location of the manufacturing facilities and the disposal of their waste systems [32]. High levels of strontium have harmful effects on humans; it may cause bone development issues for kids who consume foods that contain extremely high levels of Sr. Strontium (Sr) occurs naturally in varying amounts; the average level found in sediment samples taken from different rivers was approximately 273 mg/kg. The globally reported experience Sr content of soils varies between 130 to 240 mg/kg [33].

CONCLUSIONS

The contamination assessment of Khirbet al-Samra sediments utilized several pollution indicators including the PLI, EF, and I_{geo} . Additionally, correlation coefficient analysis was conducted on concentrations of total organic matter (TOM), Pb, Cd, Cu, and Mn, revealing varying correlations that indicate complex geochemical behaviors among these elements. The I_{geo} analysis results

indicated that concentrations of Mn, Pb, and Cu are within safe levels and are largely unaffected by anthropogenic influences in Khirbet al-Samra sediments. However, the concentration of Cd exceeded the average value, suggesting a potential risk due to pollution. Elevated Cd concentrations can be attributed to anthropogenic sources such as phosphate fertilizers and pesticides used in agricultural lands in the area, as well as the agricultural use of sewage sludge and seepage of domestic wastewater from cesspits in nearby villages. Natural weathering of surrounding geological formations also contributes to Cd contamination. The PLI analysis further confirmed that Khirbet al-Samra is at risk of environmental pollution, particularly concerning heavy metals like Cd, if these external sources are not controlled. The EF results showed high concentrations of Cu and Cd in the study area. Cd concentrations increased in some samples due to the use of cadmium-containing raw phosphate in fertilizer production and seepage of domestic wastewater from nearby villages. Cu pollution sources in some samples were attributed to agricultural practices involving sewage sludge and agro-chemicals like phosphate fertilizers and pesticides. Additionally, the proliferation of septic tanks in residential areas and seepage from cargo tanks contributed to Cu pollution. The variation in Mn concentration across the study area was linked to irrigation practices using industrial wastewater, other agronomic activities, and the presence of high organic matter content. Overall, these findings highlight the importance of monitoring and controlling anthropogenic

sources of heavy metal pollution to prevent environmental degradation in Khirbet al-Samra and similar regions.

Heavy metals (Cd, Cu, Pb, and Mn) (whose concentrations range in low levels and are not harmful to general health) have been found in the surface soil (sediments) from Khirbit al Samrah. However, it is still crucial to keep an eye on and assess the amount of heavy metals in surface soil (sediments). Monitoring its effects on the environment and general health is crucial. Use additional methods to get rid of the harmful heavy metals (Cd, Cu, Pb, and Mn) if concentrations are present.

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