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Evaluation of Influencing Factors and Potential Ecological Risks by Accumulation of Heavy Elements in Agricultural Soil Samples, Wadi Turabah (KSA)

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Abstract

Several studies have proven that heavy elements (HEs) are environmental pollutants that threaten human health and the natural environment. They affect soil quality and can be transmitted to the human body while eating foods contaminated with HEs. Intensive agriculture leads to the accumulation of fertilizer and pesticide compounds due to the excessive use of agrochemicals (ACs) to achieve better yields. However, their use leads to the loss of the authenticity of food sources with bioaccumulation of HEs. This study first aimed to determine the effect of ACs on agricultural soil samples (ASs), then, to evaluate the degree of pollution and the risks posed by HEs to the soil due to the use of ACs and other human activities. These assumptions were verified by analyzing ASs collected from specific farms in Wadi Turabah and testing them for Pb, As, and Al contents. These were proven by evaluating some pollution indicators and influencing factors such as pollution load index (*PLI*), geographic accumulation index ($I_{geo.}$), and contamination factor (CF_i). In addition to, the potential ecological risk index (E_i) and potential ecological risk intensity (*RI*). The concentrations of Pb, As, and Al in ASs varied between 0.018–0.079, 0.028–0.132, and 1.229–2.270 mg.kg^{-1}, respectively. Estimated environmental risk values indicated that the ASs were moderately contaminated by Al, with low and acceptable risks due to As and Pb, respectively. Considering the strength and severity of the toxic effect of HEs in ASs study areas, it was classified as moderate. The results of this study can be applied in taking the necessary measurements to reduce the use of ACs that may lead to environmental risk due to HEs contamination.

Keywords: Heavy elements, Agricultural soil samples, Agrochemicals, Pollution indicators, Influencing factors.

Introduction

Heavy elements (HEs) are a general term usually used to describe a group of elements with higher molecular weight and relatively high density and are toxic even at ppb levels. These elements are considered major environmental pollutants and they have harmful effects on living organisms [1]. Lead (Pb) is a highly toxic heavy element and its widespread use has caused widespread environmental pollution and health problems [2]. Arsenic (As) is an extremely toxic metalloid that is extensively distributed resulting in a variety of toxicity and harmful effects on humans and environmental health [3]. Aluminum (Al) is the most abundant and widely used metallic element in the earth's crust. It is widely distributed in nature and is the most used nonferrous element. The significant accumulation of Al in the universe led to major environmental and health problems. Although As and Al are not classified as HEs in environmental studies, but due to their toxic effects, they are listed as HEs on some toxin lists [4]. It has been found that the bioaccumulation of HEs in ASs for long periods becomes pollutants and of course, will negatively affect the food chain and human health [5]. Environmental risks were represented by a group of chemical compounds that belong to ACs. Soil contamination with these compounds has become a major environmental problem [6−8]. It poses a threat to agricultural production worldwide, due to its non-biodegradable nature and long biological half-life [2, 9].

HEs were classified as essential and/or nonessential elements based on their roles in various physiological and biochemical functions. The essential elements like Cu, Zn, and Fe, are required for living organisms, even in small quantities. While, elements like Cd, Pb, and Hg with unknown vital importance or purpose are considered non-essential and/or toxic elements [10]. Some important biological elements are useful for plant growth, as well as for the production and functioning of various biomolecules such as chlorophyll and carbohydrates [11]. Some diseases and/or deformities may result from deficiency or excess of important health HEs. Therefore, it indicates that the element may need a specific organism but not another [5, 10]. Natural sources of HEs mainly include soil erosion, geological weathering, and airborne dust [12]. Moreover, various industrial human activities are the main cause of environmental pollution [12, 13]. HEs usually undergo multiple biochemical cycles and finally enter the food chain, leading to bioaccumulation and thus threatening human health [14−16]. It has been found that environmental pollution by HEs is a widespread global problem and has caused increasing concern due to its harmful impact on human health [15]. Therefore, it was necessary to estimate the concentrations of HEs in ASs to protect the and the environment to preserve human health [17].

Overall, ASs has been recognized as a major component of the environment that has been frequently detected to accumulate HEs [18, 19]. Furthermore, ASs may be contaminated with HEs by treated wastewater, mining, emissions from industrial areas, leaded gasoline, paints, etc [20−22]. Contaminated foods with HEs may decrease immunity, delayed growth, and other diseases associated with malnutrition, cancers, etc. Also, HEs can be transmitted to the human body through food chain pathways or by direct skin contact and accumulate in various body organs [23, 24]. Moreover, the frequent use of ACs may lead to the accumulation of HEs in various plant tissues, which reduces their nutritional value [20, 25]. In addition, source identification, environmental risk assessment, and health risk assessment provide a broader assessment of pollution in the natural ecosystem [26−28]. In such studies, indicators such as PLI, $I_{geo.}$, CF_i, E_i, and RI are usually used to evaluate environmental pollution and potential ecological risks caused by HEs. Therefore, one of the main objectives of this study was to determine the degree of pollution and the toxic risks posed by various pollutants like ACs in ASs in Wadi Turabah. This study also emphasizes the need for soil management practices and environmental health. This is to reduce HEs contamination for food safety and environmental control.

Materials and Methods

Sample Collections

Mainly three areas of ASs were randomly selected in Wadi Turabah (KSA) for this study during the period from April to May 2022. Fig. 1 shows the studied area and ASs collection sites. Approximately, 150 g of each ASs were taken from the surface,10.0 cm, and 25.0 cm depth separately and well mixed [5, 29]. The specified depths have been considered as standard in soil sampling protocols to evaluate the bioaccumulation of HEs in ASs in most environmental studies. For the HEs analysis in the supernatant solution of all collected samples, the ICP technique was used. Regardless of the location and method of sample collection, a questionnaire survey was conducted to obtain information about different agricultural practices. From these questionnaires, it is possible to identify wrong agriculture practices that can be corrected through guidance and training on the usage of ACs.

Figure 1. **The locations of ASs sampling sites in Wadi Turabah, KSA**

Sample Pretreatments

Digestions of ASs were performed using a microwave digestion oven (MARS-5: Microwave Accelerated Reaction System with 5 units) before determining HEs concentration. Samples were carefully weighed and then placed in a microwave Teflon digestion vessel. About 2.0 and 4.0 mL of H_2O_2 and HNO_3, respectively, were added to each sample. The contents of the digestion vessels were then carefully shaken, then closed well and the ideal heating programs were followed. After completion of the digestion process, the contents were quantitatively transferred to a (50.0 mL) volumetric flask. Then it was diluted with distilled water to the mark. This procedure was almost the same as the procedure reported in our previous work with some very minor modifications [30]. Several analytical blanks were prepared, in the same way as the core samples for instrumental drift characterization. The ICP technique was also used to analyze standard and supernatant solutions for all collected samples.

The Analytical Figure of Merits

HEs (Pb, As, and Al) were analyzed, and then the accuracy of the analytical method was controlled using reagent blanks. The limit of detection (LODs) of Pb, As, and Al were 0.0062, 0.0054, and 0.0557 $mg.kg^{-1}$, respectively. While, the limit of quantification (LOQs) were 0.085, 0.017, and 0.175 $mg.kg^{-1}$, respectively.

Assessment of Influential Factors

To determine the magnitude of HEs contamination in the ASs and to assess the influential factors, the ecological risks in Wadi Turabah some indices were calculated. These indices include *PLI*, $I_{geo.}$, CF_i, *Ei*, and *RI*.

$I_{geo.}$ is a common measurement usually used to calculate the HEs contamination level in the ASs by averages of comparing HEs content in ASs with the geochemical background values (GBV). $I_{geo.}$ was calculated using the formula (1) [5, 27, 31, 32].

$$I_{geo.} = \log_2\left[\left(\frac{Cn}{1.5}\right)\right] xBn \qquad (1)$$

Whereas, *Cn* and *Bn* were the measured concentration and the GBV of element n in the ASs, respectively. In this study, the background geochemical compositions of non-studied ASs were selected as the GBV for calculating $I_{geo.}$ values. Therefore, a factor of 1.5 was used to account for any possible changes in the background data attributable to MARS-5. In addition to, logical variations and to minimize possible variations resulting from lithogenic source effects [5, 31]. Hakanson [33], suggested that CF_i, as a single index, is a simple and effective tool for monitoring HEs contamination. It's the ratio obtained by dividing the content of each element in ASs by the GBV to evaluate the contamination of ASs. The CF_i was calculated using the formula (2) [5, 17, 27, 31, 32, 34].

$$CF_i = \frac{C_i}{C_b} \qquad (2)$$

Since, CF_i is a contamination factor of HEs in ASs, C_i and C_b were the measured average concentration and the element in ASs, respectively.

Furthermore, *PLI* was established by the procedure of Tomlinson et al. [35]. It was used to assess the comprehensive level of HEs for ASs. *PLI* was calculated using the formula (3) [5, 27, 31, 32].

$$PLI = \sqrt[1/n]{CF_1 x CF_2 x \ldots CF_n} \qquad (3)$$

Whereas n is the number of elements, CF_1, CF_2 and CF_n are the contamination factor of elements 1, 2, and n in ASs respectively/GBV of the elements.

Moreover, E_i was proposed by Hakanson [33], *Ei* is used to evaluate the toxic risk of each HEs in ASs. *Ei* was calculated using the formula (4) [5, 27, 31, 32].

$$E_i = T_I \, X \, {C_i}/{C_o} \quad (4)$$

Since, T_i is the toxic response factor for a particular element; they were 5, 5, and 30 for Pb, As, and Al, respectively. According to the unified and standardized toxic response factor proposed by Hakanson [33], *Ci* is the element content in the ASs and *Co* is the regional background value (RBV) of each element in the ASs.

Furthermore, *RI* is a comprehensive relationship or method that links all HEs with their toxicological effects. *RI* was calculated using the formula (5) [5, 27, 31, 32].

$$RI = \sum_{i=1}^{n} Ei \quad (5)$$

Whereas E_i is the potential ecological risk index for a given element in ASs. They were adjusted to accommodate memory effects from a specific sample such as organic components and/or TDS.

Results and Discussion

ICP parameters Optimization

The emission intensity of ICP is mostly affected by radio frequency (RF-incident power, W), Ar-gas nebulizer flow rate (L min^{-1}), and sample uptake flow rate (mL min^{-1}). While plasma and auxiliary Ar-gas flow rate (L min^{-1}) and frequency (MHz) have relatively few effects on the sensitivity. They were adjusted to accommodate memory effects resulting, from a specific sample, such as organic components and/or TDS [36]. The RF-incident power (W) was studied in the range 1400−1800 W. Analysis results for almost all elements indicate that sensitivity and linearity were better at 1600 W (Table 1).

Furthermore, the effect of the Ar-gas flow rate in the nebulizer between 0.40−0.80 L min^{-1} was studied. The maximum intensity of 0.60 L min^{-1} was observed for almost all elements, thus throughout this study, a flow rate of 0.60 L min^{-1} of the Ar-gas nebulizer was adopted (Table 1). Furthermore, the sample uptake flow rate was examined at three levels of 1.0, 2.0 and 3.0 mL min^{-1}. It was found that the emission intensity of both Pb and As was higher at 1.0 mL min^{-1}, while the emission intensity of Al was slightly higher at 2.0 mL min^{-1}. Therefore, a sample uptake flow rate of 1.0 mL min^{-1} was chosen for this study, which provides low sample consumption and sufficient sensitivity (Table 1). All studied elements were measured in two different spectral emission lines that were ionic and atomic lines [36]. The selection criteria and preference between them depended on the sensitivity, spectral interferences and concentration range of each element. The specific selected line (nm) for each analyte is indicated in Table 1.

Table 1. **ICP−OES optimized conditions.**

Parameters	Selected values
RF-incident power[a] (W)	1600
Frequency (MHz)	40.68
Auxiliary Ar-gas flow rate (L min^{-1})	0.2, Ar-gas
Nebulizer Ar-gas flow rate[a] (L min^{-1})	0.60, Ar-gas
Plasma Ar-gas flow rate (L min^{-1})	15.0, Ar-gas
Sample uptake flow rate[a] (mL min^{-1})	1.0
Wavelengths[a] (nm): Pb, As and Al	220.353, 188.979, 308.212, respectively

[a] Optimized values

Microwave Parameters Optimization

Since the efficiency of sample digestion depends on the sample matrix, it's important, to optimize MARS-5 conditions. MARS-5 temperature and the digestion mixture further affect sample digestion. However, ventilation time, pressure, holding time, and ramp time have relatively minor effects on sample digestibility. The MARS-5 was set between 210–260 ºC. To select the suitable oven temperature, it was observed that clear solutions existed at 250 °C, therefore, this temperature was used in the study. Furthermore, the oxidant/acid mixture of H_2O_2/HNO_3 has been studied in a ratio of 1:1, 1:2, 1:3, 1:4 and 1:5 and a clear solution was observed in a ratio of 1:2. Therefore, a ratio 1:2, was used to digest plant samples throughout this work. While an acid/oxidizing mixture (H_2O_2/HCl/HF/ HNO_3) in a ratio of 1:1:2:7, 1:2:4:8, 1:2:3:9, 1:3:5:9 and 1:4:6:10 was studied. A clear solution was observed in the ratio of 1:2:3:9, therefore, this ratio was used to digest ASs in this study. The optimal values of MARS-5 parameters are shown in Table 2.

Table 2. **MARS-5 heating program for digestion of ASs.**

Conditions	Selected values
Ramp time (min)	25
Ventilation (min)	10
Holding time (min)	10
Pressure (PSI)	800
Temperature[a] (°C)	250

[a] Optimized values

The Analytical Figures of Merits

The specific wavelengths (nm) that give maximum emission intensities and high sensitivities under optimal operating conditions for the ICP are described in Table 1. Furthermore, the linearity of the method was tested using the selected analytical line and was determined at five concentrations in the range of 0.04−100 $mg.L^{-1}$. This was satisfactory for all studied elements in which the R^2 value was higher than 0.9992 in the linear regression curve. This value confirms the linearity of the selected analytical method by the standards set by the AOAC [37]. The accuracy of the analytical method was determined by calculating the recovery (%). In this study, it was found within the acceptable range for all analytes [100 ± 6] (Table 3). This is good and indicates, that there were no significant gains or losses for analytes using the developed analytical method.

In addition, the precision of the ICP method was calculated as RSDs%. This was done from five independent replicates for each sample and was found to be < 3.17% (Table 3). This value confirms the precision of the analytical method used. The LODs ($mg.kg^{-1}$) of the analytes ranged between 0.0054–0.0557 $mg.kg^{-1}$ for Cd and Al, respectively, while the LOQs ($mg.kg^{-1}$) ranged between 0.017 $mg.L^{-1}$ for As and 0.175 $mg.L^{-1}$ for Al. The obtained values (LODs and LOQs), clearly demonstrated the high sensitivity and linear range of ICP-method (Table 3).

Table 3. **The analytical figure of merits values for the determination of HEs in ASs.**

HEs	R^2	Recoveries (%)[a]	RSDs (%)	LODs ($mg.kg^{-1}$)	LOQs ($mg.kg^{-1}$)
Pb	0.9998	98±4	2.98	0.0062	0.085
As	0.9998	102±5	1.02	0.0054	0.017
Al	0.9993	106±3	3.16	0.0557	0.175

[a]Recoveries (%) were expressed as mean±SD

HEs Contents of ASs

HEs is a general term used to describe a group of elements with high density and high molecular weight. In general, these elements are considered major environmental pollutants, even if they are found in small quantities [1]. According to the survey conducted among the local farmers, natural fertilizers such as goat dung, urea, and ammonia were almost applied in the lands. Moreover, during the germination and growth periods, they used ACs like dichlorodiphenyltrichloroethane (DDT) and dichlorodiphenyldichloroethane (DDD).

The concentrations of HEs were studied in the ASs in studied areas in Wadi Turabah (KSA) and their values were reported in Table 4. The concentrations of Pb in ASs in the studied area were in the range of 0.018–0.079 $mg.kg^{-1}$. The average Pb content in ASs was 0.049 $mg.kg^{-1}$ (Table 4), while the concentration of As in ASs in studied areas was in the range of 0.028–0.132 $mg.kg^{-1}$. The average As content in ASs was 0.080 $mg.kg^{-1}$ (Table 4). Moreover, the concentrations of Al in ASs in studied areas were in the range of 1.229–2.270 $mg.kg^{-1}$. The average Al content in ASs was 1.750 $mg.kg^{-1}$ (Table 4). Foliar application of fertilizers and pesticides in ASs is slightly increased in Pb and As contents and highly increasing in Al content. Despite that HEs contents in the ASs in studied areas were found at acceptable levels (Table 4).

Table 4. **Concentrations of HEs in ASs in studied areas.**

Samples	Levels	Conc. of HEs ($mg.kg^{-1}$)		
		Pb	As	Al
Ass	Min.	0.018	0.028	1.229
	Max.	0.079	0.132	2.270
	Average	0.049	0.080	1.750

Determination of ASs Contamination by HEs

The influential factor in ASs of studied areas includes surveys were calculated and the values were presented in Tables 5, Fig. 2, 3, 4, and 5.

Table 5. **The influential factors assessment and GBV of elements represent the average contents in ASs of studied areas.**

Elements/average values	Average values		
	Pb	As	Al
PLI	0.0028	0.0057	6.6340
$I_{geo.}$	0.0963	0.0889	3.5370
CF_i	0.005	0.014	1.053
E_i	6.310	5.140	22.76
GBV around the study areas [32]	9.880	5.530	10.07

PLI is an effective tool in assessing environmental pollution by HEs, and it has been used to evaluate the comprehensive level HEs in ASs [35]. According to Chakravarty and Patgiri [38], the *PLI* value > 1 means polluted while *PLI* value < 1 indicates unpolluted. Calculated *PLI* in ASs was < 1 therefore, it was classified the toxicological effects of HEs of ASs as moderate degree based on their intensities. If the GBS of pollutants were present, *PLI* value would be less than the value one (i.e., < 1). But when *PLI* exceeds the value one (i.e., > 1) it confirms the deterioration of ASs quality [5].

$I_{geo.}$ is a common measurement tool, used to calculate the HEs contamination level in ASs. The factor of 1.5 value is used to minimize as much as possible variations due to lithogenic effects. $I_{geo.}$ values were calculated based on HEs contents in the studied ASs. The results showed that there were no obvious signs of contamination, which most likely indicates the geological origin of the elements in the ASs (Table 4). Kumar and Pratap [31], proposed seven classes (grades or scores) of $I_{geo.}$ index values of enrichment for contamination level in ASs, which were summarized in Table 6.

When considering the recognized seven scores (from class 0 to 6) with the results of study areas for the ASs were uncontaminated to strongly contaminated by studied HEs. It's found that $I_{geo.}$ value of Pb shows the land is uncontaminated to moderately contaminated; $I_{geo.}$ values show it is moderately to strongly contaminated by As and Al, respectively. The higher degree of pollution for Al and As was associated with the agricultural and/or other anthropogenic activities in the studied areas.

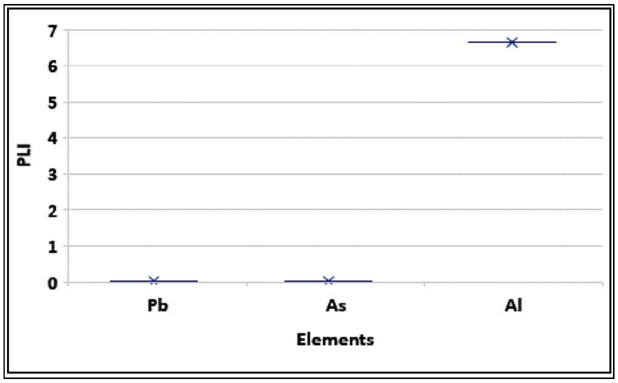

Figure 2. ***PLI*** **values of HEs in the ASs from Wadi Turabah**

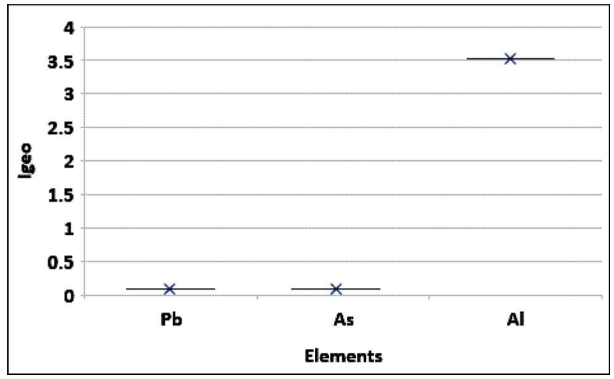

Figure 3. **$I_{geo.}$ values of HEs in the ASs from Wadi Turabah**

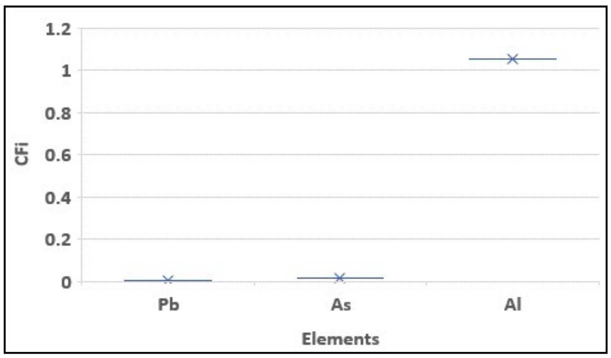

Figure 4. **CF_i values of HEs in the ASs from Wadi Turabah**

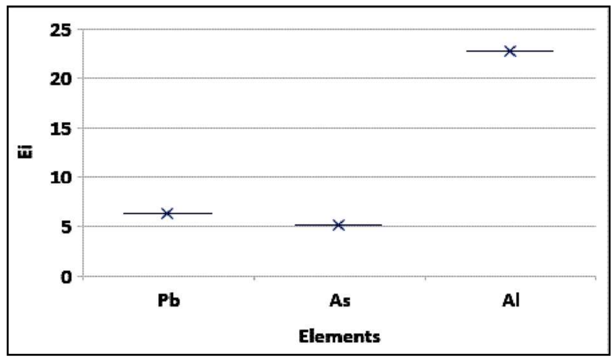

Figure 5. **E_i values of HEs in the ASs from Wadi Turabah**

Table 6. **Standard $I_{geo.}$ for contamination levels in ASs that recognized by Subramaniam, et al.** [5], **Kumar and Pratap** [31], **and Birch** [39].

Classes	I_{geo}. values	Contamination levels
Class-0	$I_{geo.} < 0$	Practically unpolluted (uncontaminated)
Class-1	$0 < I_{geo.} < 1$	Varied between unpolluted (uncontaminated) to moderately polluted (moderately contaminated)
Class-2	$1 < I_{geo.} < 2$	Moderately polluted (moderately contaminated)
Class-3	$2 < I_{geo.} < 3$	Varied from moderately (moderately contaminated) to strongly polluted (strongly contaminated)
Class-4	$3 < I_{geo.} < 4$	Strongly polluted (strongly contaminated)
Class-5	$4 < I_{geo.} < 5$	Varied between strongly to extremely polluted (strongly to extremely contaminated)
Class-6	$I_{geo.} > 5$	Extremely polluted (extremely contaminated)

Moreover, CF_i is a contamination factor used to assess the ASs contamination by HEs in ASs in studied areas. The classes of CF_i for ASs are summarized in Table 7. The $I_{geo.}$ values calculated based on the concentrations of elements in the studied ASs do not reveal any clear sign of contamination. This likely indicates the geological origin of the elements in these ASs (Table 4).

Table 7. **The classes of CF_i proposed by Hakanson and others [33, 35].**

Classes	*CFi* values	Toxic risk of HEs
Class-0	$CF_i \leq 1$	Practically low or uncontaminated (practically low or unpolluted)
Class-1	$1 \leq CF_i < 3$	Moderately contaminated (moderately polluted)
Class-2	$3 \leq CF_i < 6$	Considerably contaminated (considerably polluted)
Class-3	$CF_i \geq 6$	Very highly contaminated (very highly polluted)

Furthermore, the potential ecological risk index (E_i) and classification of risk intensity were classified by Hakanson [33], into five (5) grades ranging from ≤ 40 to ≥ 320, E_i levels values were indicated in Table 8.

Table 8. **Classification of *Ei* values according to the standardized toxic response factor proposed by Hakanson and others** [33, 35]**.**

Classes	E_i values	Toxic risk of HEs
Class-0	$E_i \leq 40$	Practically low or uncontaminated (practically low or unpolluted)
Class-1	$40 \leq E_i < 80$	Moderately contaminated (moderately polluted)
Class-2	$80 \leq E_i < 160$	Highly contaminated (highly polluted)
Class-3	$160 \leq E_i < 320$	Very highly contaminated (very highly polluted)
Class-4	$E_i \geq 320$	Dangerous contaminated (dangerous polluted)

The calculated potential ecological index (E_i) of studied HEs (Pb, As, and Al) was less than 40 (Table 9). These recognized five scores (from class 0 to 4) which indicated that ASs were practically low or uncontaminated by studied HEs. The levels of studied HEs in the studied ASs were categorized as potentially low risk.

Table 9. **Results of potential ecological risk index (*E_i*).**

Element	T_i	C_i	C_o	E_i
Pb	5.0	12.47	9.88	6.31
As	5.0	5.7	4.54	5.124
Al	30.0	0.22	0.29	22.76

T_i is the toxic response factor for a given element, C_i is the element content in the ASs, and C_o is the RBV of any elements in the ASs, E_i is the potential ecological risk index

RI values are a comprehensive method that relates all HEs to their toxicological effects. Their values may be classified based on their intensities and according to their toxicological effects proposed by Hakanson and others [33, 35], on a scale ranging from 150 to 600 were indicated in Table 10.

Table 10. **Classification of *RI* values according to the toxicological effects proposed by Hakanson and others** [33, 35]**.**

Classes	RI values	Toxicological effects of HEs
Class-0	$RI \leq 150$	Practically low or uncontaminated (practically low or unpolluted)
Class-1	$150 \leq RI < 300$	Moderate degree contaminated (moderate degree polluted)
Class-2	$300 \leq RI < 600$	Considerable high degree contaminated (considerably high degree polluted)
Class-3	$RI \geq 600$	Very high degree contaminated (very high degree polluted)

According to the calculated RI values (Table 10) in the studied areas was 160, therefore, the toxicological effects of HEs on ASs were exhibited at a moderate degree according to their intensities

Conclusion

The presence of HEs in ASs was a challenging issue associated with potential ecological risks to human health. The results of this study revealed the presence of Al, Pb, and As in ASs, Wadi Turabah (KSA). The results of this research indicate that the contents of Pb, As, and Al in ASs varied between 0.018–0.079, 0.028–0.132, and 1.229–2.270 mg.kg^{-1}, respectively. The average concentrations were increased in the order of Al > As > Pb. Some useful pollution indexes have been used to evaluate the level of pollution resulting from HES. In addition, the effect of pollutants on the quality of ASs in the study areas was studied. Based on the influential factors obtained values, the analyzed ASs were determined as uncontaminated for almost all of the studied HEs. The assessment of pollution identified, as the HEs with significant contribution to environmental pollution due to the continuous usage of ACs and the impacts of anthropogenic activities in the study areas. Considering various potential ecological risks indices, it was found that the ASs was uncontaminated by Pb and moderately contaminated by As and Al, respectively in the order Al > As > Pb.

Conflict of Interest

The author declares that there is no conflict of interest.

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