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RESEARCH ARTICLE

ECOLOGICAL EVALUATION OF UNCOMMON HEAVY METALS CONTAMINATION IN THE SOILS OF THE CENTRAL PROVINCE OF UŞAK, WESTERN TURKIYE

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ABSTRACT

This study aimed to evaluate the concentrations of the uncommon heavy metals (Ag, Bi, Co, Sb, Th, Tl, U, and V) in 90 soil samples obtained from both urban and agricultural lands near Uşak, western Turkiye to investigate dimensions of the existing ecological pollution using geoaccumulation and enrichment factor indices, and to identify their potential pollutants. The concentration values for the selected elements ranged from 0.01 to 0.46 mg/kg for silver (Ag); 0.07 to 0.72 mg/kg for bismuth (Bi); 7.9 to 55.8 mg/kg for cobalt (Co); 0.12 to 27.99 mg/kg antimony (Sb); 3.4 to 17.7 mg/kg for thorium (Th); 0.04 to 0.5 mg/kg for thallium (Tl); 0.3 to 7.3 mg/kg for uranium (U); and 18 to 72 mg/kg for vanadium (V). Igeo values of Ag showed moderate to heavy contamination in the city center of Uşak province. Igeo values of Bi and Sb in the west part of the study area indicated extremely contaminated soils. EF values for Bi and Sb also showed significant enrichment in the soils in the western portion of the study area which further validates that the potential sources for Bi and Sb heavy metals contaminations might be anthropogenic.

Keywords: *Uncommon heavy metals contamination, Geoaccumulation index, Enrichment Factor, Bismuth, Antimony.*

1. INTRODUCTION

Rapid industrialization and population growth, especially in developing countries, have started to be inevitable problems related to environmental pollution by heavy metals contamination which is one of the biggest issues facing the world today. Heavy metals are generally defined as metals or metalloids with a density higher than 5 g/cm^3 [1-2]. It has been observed by numerous scholars that toxic elements (e.g., heavy metals) infiltrate humans and animals via the skin, the digestive system, or respiration. [3-4]. Toxic heavy metals have been proven to pose a major threat to human and animal health because of their capacity to affect DNA and disrupt the functioning of enzymes and protein molecules [5]. Although the heavy metals enter the organism easily, they are not so easily expelled from the metabolism and accumulate in the organism over time. Most of the health problems due to heavy metals accumulation in the human metabolism are chronic diseases or cancers that require

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advanced diagnosis and treatment [6]. The heavy metals accumulation in soils occurs mostly at the surface or depths close to the surface. Almost all of the heavy metals are adsorbed by clay minerals in the soil or turn into stable forms by forming organic compounds in the soil. The heavy metal concentration in the soil decreases with depth [7], thus physicochemical properties of the soils play a major role during ecological assessment efforts. Heavy metals contamination in the soil occurs usually due to agricultural activities, waste disposal, industrial activities and effluents, mining and smelting, corrosion of metallic materials, motor vehicle exhaust, and electronic and biomedical waste (Figure 1). Environmental pollution caused by the accumulation of common heavy metals (As, Al, Cd, Cr, Cu, Hg, Pb, Ni, and Zn) and their anthropogenic sources in soils has been extensively studied before. However, the ecotoxicity of uncommon heavy metals (Ag, Bi, Co, Sb, Th, Tl, U, and V) in soils is currently underestimated and needs to be researched in order to better understand their impact on the environment [8]. This is due to the fact that their pollution is far less prevalent than that of common heavy metals. The purpose of this study is: (1) to analyze the concentrations of the selected uncommon heavy metals (Ag, Bi, Co, Sb, Th, Tl, U, and V) by obtaining chemical data from 90 samples collected from both urban and agricultural soils within the municipal boundaries of Uşak province, Turkiye; (2) to examine the extent of the current ecological pollution using a variety of geostatistical techniques, and (3) to identify their potential pollutants.

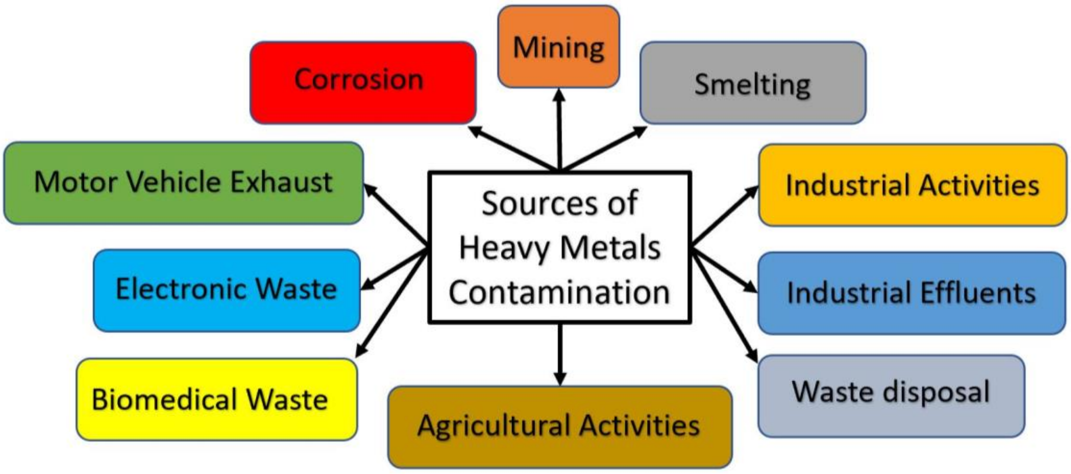

Figure 1. The image shows common anthropogenic pollutant sources of heavy metals contamination in soils.

2. STUDY AREA

The research site is situated in the central province of Uşak, a small city located in western Turkiye (Figure 2). Uşak province has a land area of around 5,341 km^2 and its total population is approximately 375,000 according to the 2021 census [9]. The climate in Uşak alternates between a continental climate, where the temperature differences between the seasons are large, and a Mediterranean climate, where summers are hot and mostly dry, and winters are generally mild. The

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annual average temperature in Uşak is 12°C and the annual precipitation varies between 430 mm and 700 mm.

Uşak province has a long history of industrialization. Turkiye's first factory processing sugar was established in 1926 in Uşak, consequently, the first electricity in Turkiye was utilized at this establishment. Uşak province received its share of the rapid industrialization, along with other provinces in Turkiye. Due to the city's strategic location along the E-96 major highway connecting Ankara and Izmir, its accessibility to international ports, and the availability of a rail transit system, industrialization has become inevitable for the city. In 1988, a mixed-organized industrial zone was established for leather tanning, and the organized industrial zone began its operations in 1994. As of 2022, the city of Uşak is home to 625 businesses, both large and small, that are currently engaged in a variety of industries, including the manufacturing of ceramics, ferrous metals, chemical paints, solid waste incineration, leather tanning, and textile. Furthermore, the population of the central province of Uşak is around 256,000, and the number of registered vehicles exceeds 149,000 [9].

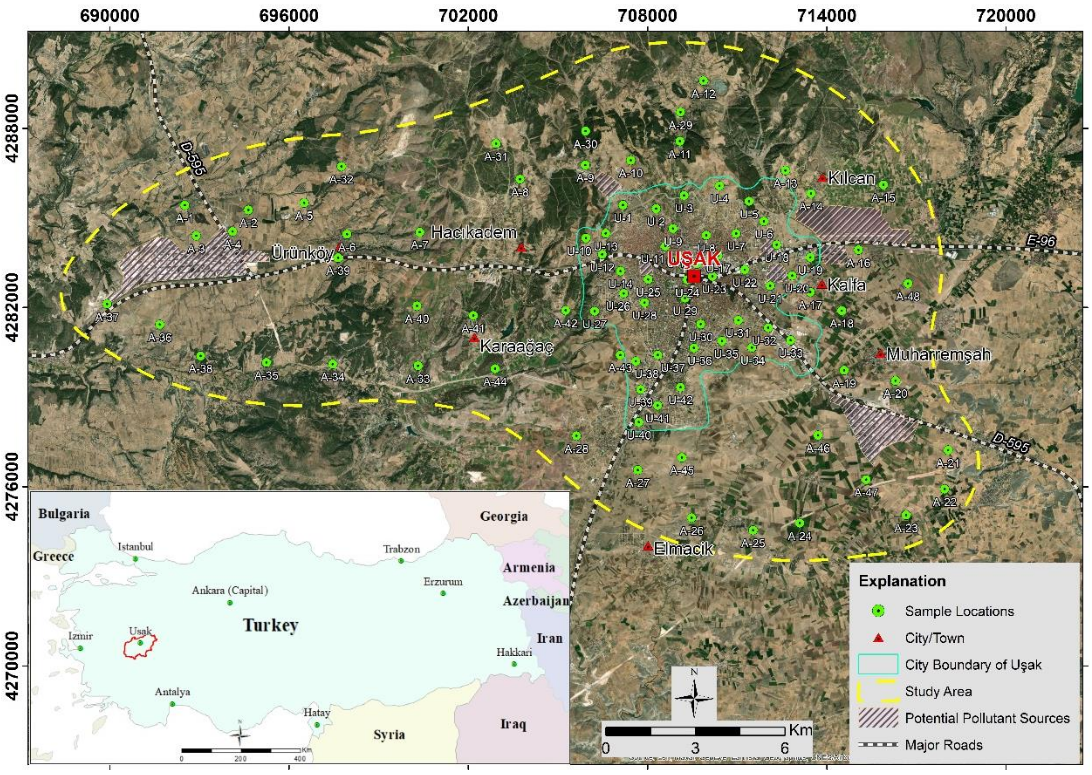

Figure 2. The study area and the soil sampling points are shown in two maps.

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2.1. Regional Geologic Settings

Paleozoic age metamorphic rocks (gneiss, schist, and marble) of the Menderes Massif form the oldest tectonostratigraphic units in Uşak [10] (Figure 3). The Jurassic Kızılcasöğüt Formation unconformably overlays the Menderes Massif, which is composed of metasandstone, metasiltstone, and slightly dolomitized limestones. A mélange, considered to have formed in an accretionary complex, was emplaced on the Kızılcasöğüt Formation in Late Cretaceous. Tertiary volcanic activities further shaped the geology of the region (Beydağ and Karaboldere formations). The Uşak region was affected by the western extensional province in the Lower Miocene. As a result, structural basins were formed during Neogene. Lacustrine argillaceous limestones (Ulubey Formation) were deposited in these basins during the Pliocene, and with the drying of these lakes during the Quaternary Period, terrestrial sediments were deposited in both alluvial and fluvial environments.

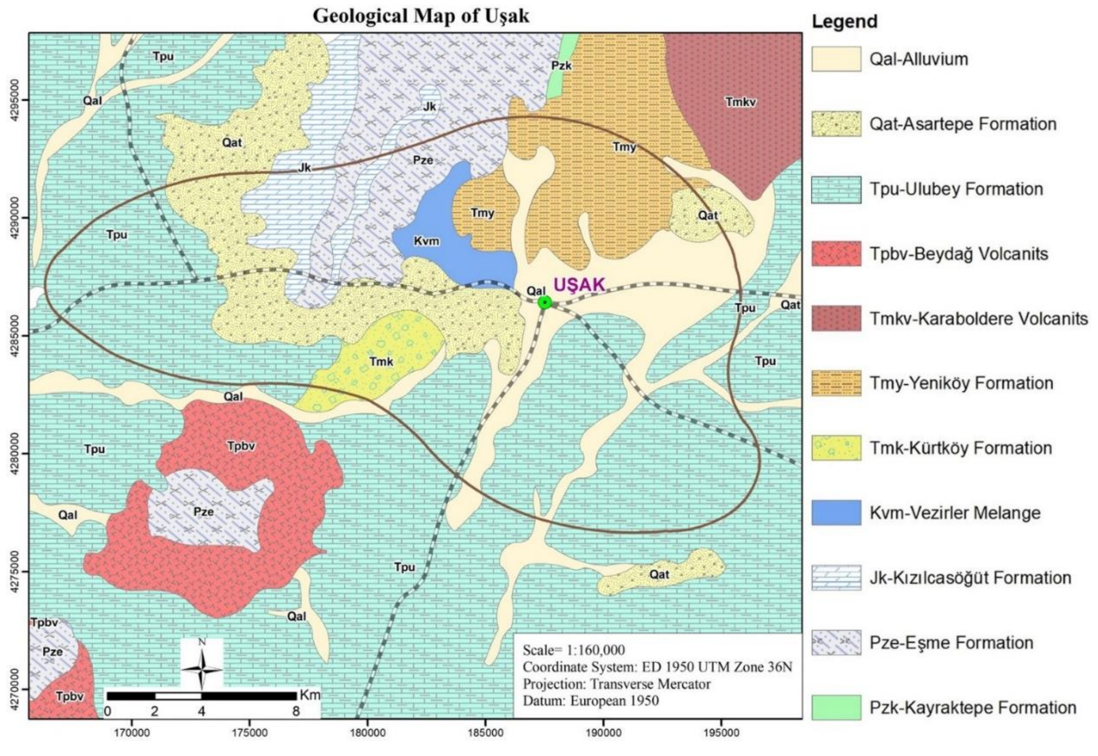

Figure 3. Geologic map of Uşak showing major formations [10].

3. MATERIALS AND METHODS

3.1. Analytical Methods

90 soil samples were taken both from urban and agricultural lands of the central province of Uşak considering the locations of potential pollutant sources. The research site covers an area of

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approximately 340 km^2 (Figure 2). Soil samples were collected in September 2021. Unique object IDs were assigned for both urban (U-1, U-2…..) and agricultural (A-1, A-2…..) soil samples. Plastic shovels were used for sampling and approximately one-kilogram soil sample was taken from 0-20 cm depth. The samples were kept until further analysis in gallon-size sealable plastic bags. In situ pH measurements were also made by using a portable Hanna pH meter. Each sampling site's geographic location was documented using a handheld Garmin E-trex unit. The coordinate system was set up using European Datum 1950 and Zone 35 North of the Universal Transverse Mercator (UTM). All of the soil samples taken from the study area were dried at room temperature for 5 days in a laboratory environment. Then soil specimens were passed through an 80-mesh sieve to obtain a grain size of 180 µm, which is ideal for chemical analysis of soil samples [11-12]. The dissolution process of the samples was carried out in Aqua Regia solution (HCl+HNO3+H2O) of 0.5 g dried and powdered soil sample at 95°C for an hour. This technique is frequently used in the chemical analysis of soils because it is one of the closest solubilization methods to the total concentrations [13]. Concentration measurements of selected uncommon heavy metals (Ag, Bi, Co, Sb, Th, Tl, U, and V) from the solutions were made by ACME (Canada) laboratories using AQ250-Ultratrace ICP-MS method. Later, the same method was used to analyze four blank soil pulps. For the purposes of quality assurance and control, the analytical procedures were tested using the standardized reference materials DS11 and STD OREAS262. In order to determine ecologic pollution levels in soils; geoaccumulation and enrichment factor parameters were utilized. Data from the analysis were then transferred to a personal computer for additional assessments. All geostatistical operations and map productions were performed using ArcMap software [14].

3.2. Data Analysis
3.2.1. Geoaccumulation index
The geoaccumulation index (Igeo) is employed to examine the level of metal pollution in the soil by comparing the existing element values with the values before industrialization. The Igeo was first introduced by Müller [15] and is currently used by many researchers for the assessment of soil pollution [16-17-18-19-20-21]. The following equation can be used to calculate geoaccumulation (Eq.1):

$$\text{Igeo} = \log_2\left(\frac{\text{Cn}}{1.5\text{xBn}}\right) \quad (1)$$

where Cn is the value of the metal concentration in the soil sample; Bn is the mean value of n metal in soil [22]; 1.5 is the base value matrix corresponding to correlation factor values. Müller [15] divided geoaccumulation index values into 7 different pollution categories:

Category	**Value**	**Soil quality**
i	Igeo ≤ 0	Unpolluted
ii	0<Igeo < 1	Unpolluted to moderately polluted
iii	1<Igeo < 2	Moderately polluted
iv	2<Igeo < 3	Moderately to heavily polluted
v	3<Igeo < 4	Heavily polluted
vi	4<Igeo < 5	Heavily to extremely polluted

vii	5<Igeo	Extremely polluted

3.2.2. Enrichment factor

The enrichment factor (EF) is a method that is widely used to identify and compare the rate and degree of soil pollution associated with heavy metals [23]. The EF is determined by normalizing the amount of a measured element to the reference value of the same element. The reference element is characterized by the fact that its concentration in the soil does not change and it has a very low chemical reaction [24-25]. Mn, Al, Fe, Zn, Ti, and Sc are the reference elements that are most commonly utilized [25-26-27-28-29-30]. During this research, Mn was employed as the normalizing reference element. For EF calculations, Mielke’s [22] composition of the Earth's crust was used for reference values of all studied elements (Ag: 0.075 mg/kg; Bi: 0.0085 mg/kg; Co:25 mg/kg; Sb:0.2 mg/kg; Th: 9.6 mg/kg; Tl: 0.85 mg/kg; U: 2.7 mg/kg; V: 120 mg/kg). The EF can be calculated with Eq. 2:

$$EF=\frac{\left(\frac{C_n}{C_{ref}}\right)\quad sample}{\left(\frac{B_n}{B_{ref}}\right)\quad background} \qquad (2)$$

where Cn is the quantity of the studied element in the soil sample, Cref is the quantity of the reference element in the studied sample, and Bn is the quantity of the examined element in the reference environment whereas Bref is the quantity of the reference element in the environment that serves as the reference [21, 22, 25]. Sutherland [30] divided EF values into 5 classes:

Class	**EF value**	**EF category**
i	EF < 2	Minimal enrichment
ii	EF = 2–5	Moderate enrichment
iii	EF = 5–20	Significant enrichment
iv	EF = 20–40	Very high enrichment
v	EF > 40	Extremely high enrichment

4. RESULTS

Descriptive statistical results of the analyses of 90 soil samples taken from the central province of Uşak are presented in Table 1. Heavy metals concentrations obtained from 90 soil samples were used to investigate potential ecological pollution within the study area. Igeo values are given in Table 2, and EF values are presented in Table 3.

4.1. Concentrations of Heavy Metals

The uncommon heavy metals concentration values for the selected elements ranged from 0.01 to 0.46 mg/kg (average: 0.04 mg/kg) for silver (Ag); 0.07 to 0.72 mg/kg (average: 0.25 mg/kg) for bismuth (Bi); 7.9 to 55.8 mg/kg (average: 23.5) for cobalt (Co); 0.12 to 27.99 mg/kg (average: 1.04 mg/kg) for antimony (Sb); 3.4 to 17.7 mg/kg (average: 8.3 mg/kg) for thorium (Th); 0.04 and 0.5 mg/kg (average: 0.26 mg/kg) for thallium (Tl); 0.3 to 7.3 mg/kg (average: 0.9 mg/kg) for uranium (U); and 18 to 72

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mg/kg (average: 35.7 mg/kg) for vanadium (V). Soil Quality Standards of Turkiye (SQST) set the maximum allowable limits for cobalt, thallium, and uranium as 20 mg/kg, 5 mg/kg, and 1 mg/kg respectively [31]. Maximum permissible concentrations in soils [8] for Ag, Bi, Sb, Th, and V are 3.1 mg/kg, 6.6 mg/kg, 3.2 mg/kg, 28.5 mg/kg, and 296 mg/kg respectively. This study shows the mean concentration values of Co exceed the SQST's limit, however, Ag, Bi, Sb, Th, Tl, U, and V values appear to be in a normal range. In addition, the mean concentration values of the studied heavy metals were compared to mean concentration values from various locations in the world [32, 33, 34, 35]. According to the findings of this study, none of the mean heavy metal concentrations exceeded the results obtained from other locations in the world (Table 1). The pH of the soil samples in the central province of the Uşak varied between 7.4 and 8.5, with a mean value of 8.0, indicating alkaline-type soils. Generally, prevailing winds blow from east to west.

Table 1. Heavy metals concentrations (mg/kg) and descriptive statistics of soil samples of Uşak.

	pH	Ag	Bi	Co	Sb	Th	Tl	U	V
Mean	8.03	0.04	0.25	23.54	1.04	8.36	0.26	0.93	35.71
Median	8.04	0.03	0.24	22.85	0.59	7.50	0.25	0.70	35.00
Min	7.42	0.01	0.07	7.90	0.12	3.40	0.04	0.30	18.00
Max	8.51	0.46	0.72	55.80	27.99	17.70	0.53	7.30	72.00
Skewness	-0.52	5.46	1.71	1.32	8.62	0.77	0.53	5.34	0.78
Kurtosis	1.21	35.79	4.83	2.78	78.33	-0.20	0.01	37.69	0.84
St. Dev.	0.19	0.06	0.11	8.69	2.97	3.42	0.10	0.84	10.86
Other studies (mean)	-	3.71	2.14	30	3.5	9	0.3	2	43

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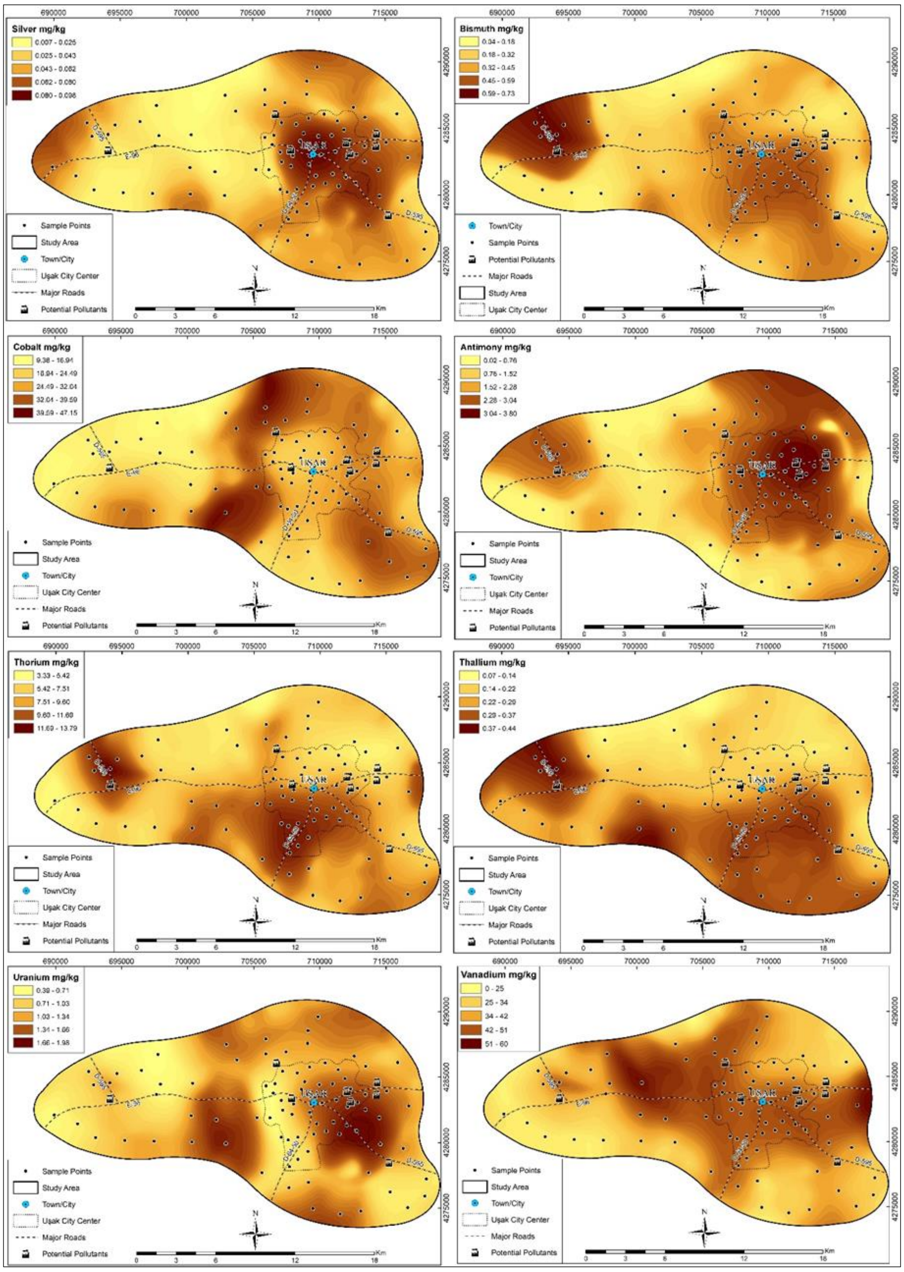

Figure 5. Spatial distribution maps show the fluctuations in the selected uncommon heavy metal concentrations.

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Furthermore, ArcMap's kriging interpolation algorithm was utilized to produce distribution maps for each heavy metal concentration (Figure 5). The Ag concentration map shows elevated values in the city center. The concentration map for Bi indicates accumulation in the northwest part of the Uşak. The Co map shows higher concentration values surrounding the city center in every direction except due south. The northeast and northwest regions of the study area, as well as the city center, show elevated values on the Sb concentration map. The Th map shows that the study region has high values in the west and south. The Tl map mimics the Th map and shows elevated concentration values in the west and the south. A map for the U concentrations shows higher values in the east part of the city as well as the south and south-central part of the study area. The V map indicates accumulation within the city center and south-central portion of the research site. The obviously elevated concentration values of Bi, Tl, Th, and Sb in the west are directly correlative with the location of the largest industrial zone in the Uşak (Figure 5).

4.2. Geoaccumulation Index (Igeo) Values

A statistical summary of the Igeo values of the selected uncommon heavy metals in 90 specimens from the central province of Uşak is shown in Table 2. Igeo maps created for the selected uncommon heavy metals are also presented in Figure 6. Igeo values for Tl and V show no contamination within the study area which may indicate the concentration values at this location is due to geogenic sources. At certain locations, Igeo values for Co, Th, and U indicate uncontaminated to moderately contaminated soils. Igeo values of Ag suggest moderate to heavy contamination in the city center of Uşak province. Bi and Sb Igeo values show extremely contaminated soils in the west, around the large industrial zone, and in the city center of Uşak (Figure 6).

Table 2. Statistical summary of the geoaccumulation index (Igeo) values for the selected uncommon heavy metals within the study area.

	I_{geo}(Ag)	I_{geo}(Bi)	I_{geo}(Co)	I_{geo}(Sb)	I_{geo}(Th)	I_{geo}(Tl)	I_{geo}(U)	I_{geo}(V)
Mean	-1.78	4.19	-0.76	0.91	-0.90	-2.43	-2.39	-2.40
Median	-1.79	4.20	-0.71	0.98	-0.94	-2.38	-2.53	-2.36
Min	-3.81	2.46	-2.25	-1.32	-2.08	-4.99	-3.75	-3.32
Max	2.04	5.82	0.57	6.54	0.30	-1.27	0.85	-1.32
Skewness	0.94	-0.14	0.01	1.15	0.11	-0.84	1.12	-0.01
Kurtosis	2.46	0.83	0.54	4.07	-0.88	2.22	2.58	-0.39
St. Dev.	0.99	0.61	0.51	1.24	0.58	0.61	0.78	0.43

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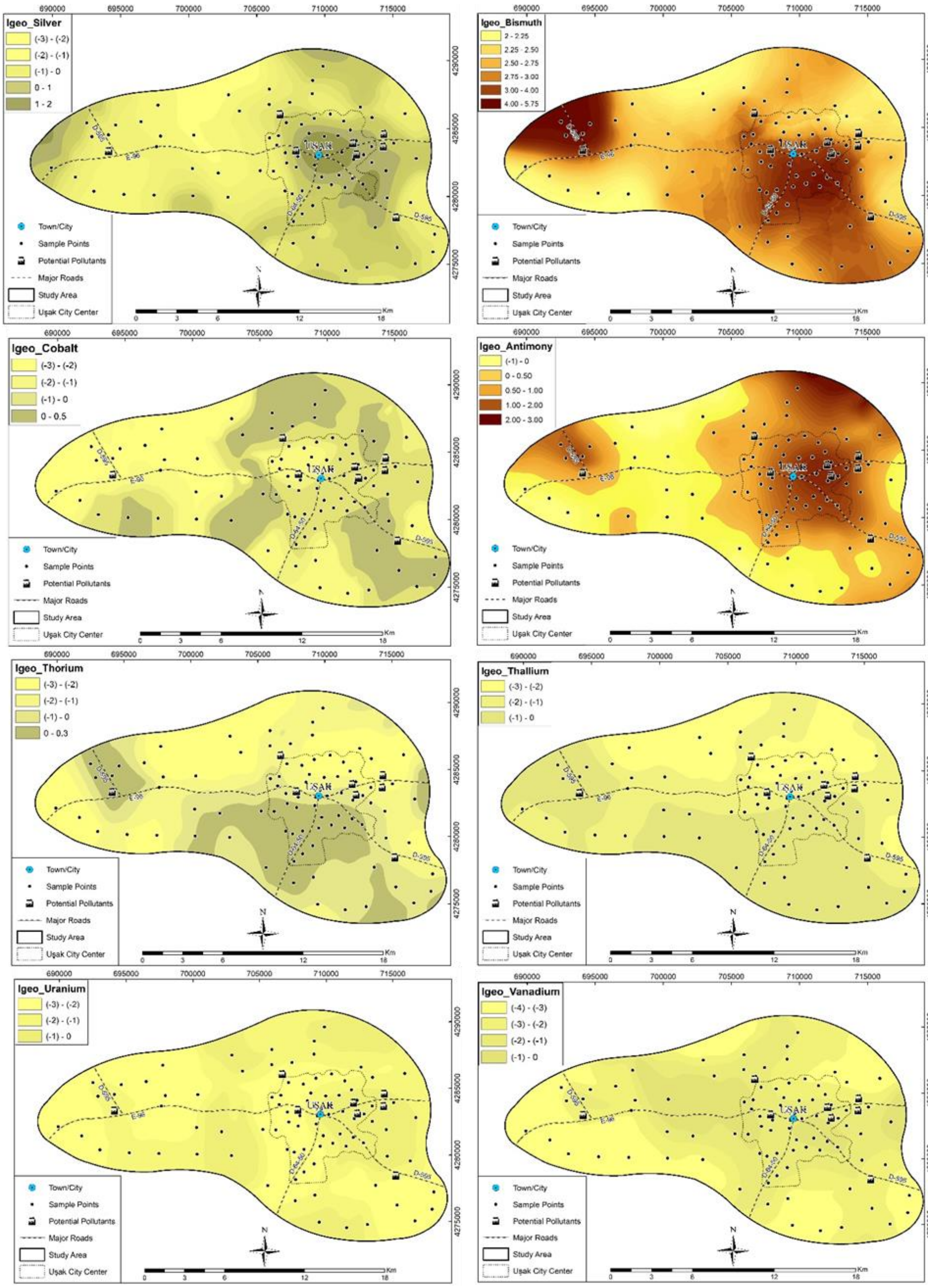

Figure 6. Maps showing the Igeo values for the uncommon heavy metals within the study area.

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4.3. Enrichment Factor (EF) Values

A statistical summary of the enrichment factor (EF) values of the uncommon heavy metals in 90 soil samples from the central province of Uşak is presented in Table 3. The following are the mean EF values, listed from highest to lowest: Bi (51.62) is followed by Sb (8.82), Co (1.58), Th (1.53), Ag (1.01), U (0.65), Tl (0.52), and V (0.52). EF values for Ag, Co, Th, Tl, U, and V indicate minimal heavy metals enrichment in the research site. In addition, maps were created for the EF values and are presented in Figure 7. EF values for Sb suggest significant enrichment, especially in the northeast and northwest parts of the research site, and extremely high enrichment within the city center. EF values for Bi indicate significant enrichment in the city center and extremely high enrichment in the south-central and west parts of the study area (Figure 7). According to Yan et al., [36], both natural and anthropogenic sources can release Sb and Bi into the environment. In comparison to geogenic sources, anthropogenic activities such as smelting, fuel burning, waste incineration, the production of plastics and textiles, and motor vehicle brake wear have a significant impact on elevating Sb and Bi concentrations in soils [37, 38, 39]. Presence of the city's largest industrial site in the west where elevated EF values of Sb and Bi suggests that industrial activities might be the potential sources of the pollution. Domestic waste, corrosion, and industrial activities in the city center might also be the source for the significant enrichment of these two substances (Sb and Bi).

Table 3. Statistical summary of the EF values for the selected uncommon heavy metals within the study area.

	EF(Ag)	EF(Bi)	EF(Co)	EF(Sb)	EF(Th)	EF(Tl)	EF(U)	EF(V)
Mean	1.01	51.62	1.58	8.82	1.53	0.52	0.65	0.52
Median	0.72	44.58	1.41	4.49	1.37	0.47	0.41	0.47
Min	0.15	12.01	0.50	1.20	0.42	0.06	0.13	0.15
Max	11.70	214.50	5.99	242.61	11.30	3.05	7.46	3.28
Skewness	5.96	3.31	3.88	8.66	6.75	5.47	5.96	6.50
Kurtosis	42.10	14.36	22.32	78.88	55.97	41.31	42.28	51.70
St. Dev.	1.39	30.14	0.66	25.73	1.17	0.32	0.88	0.34

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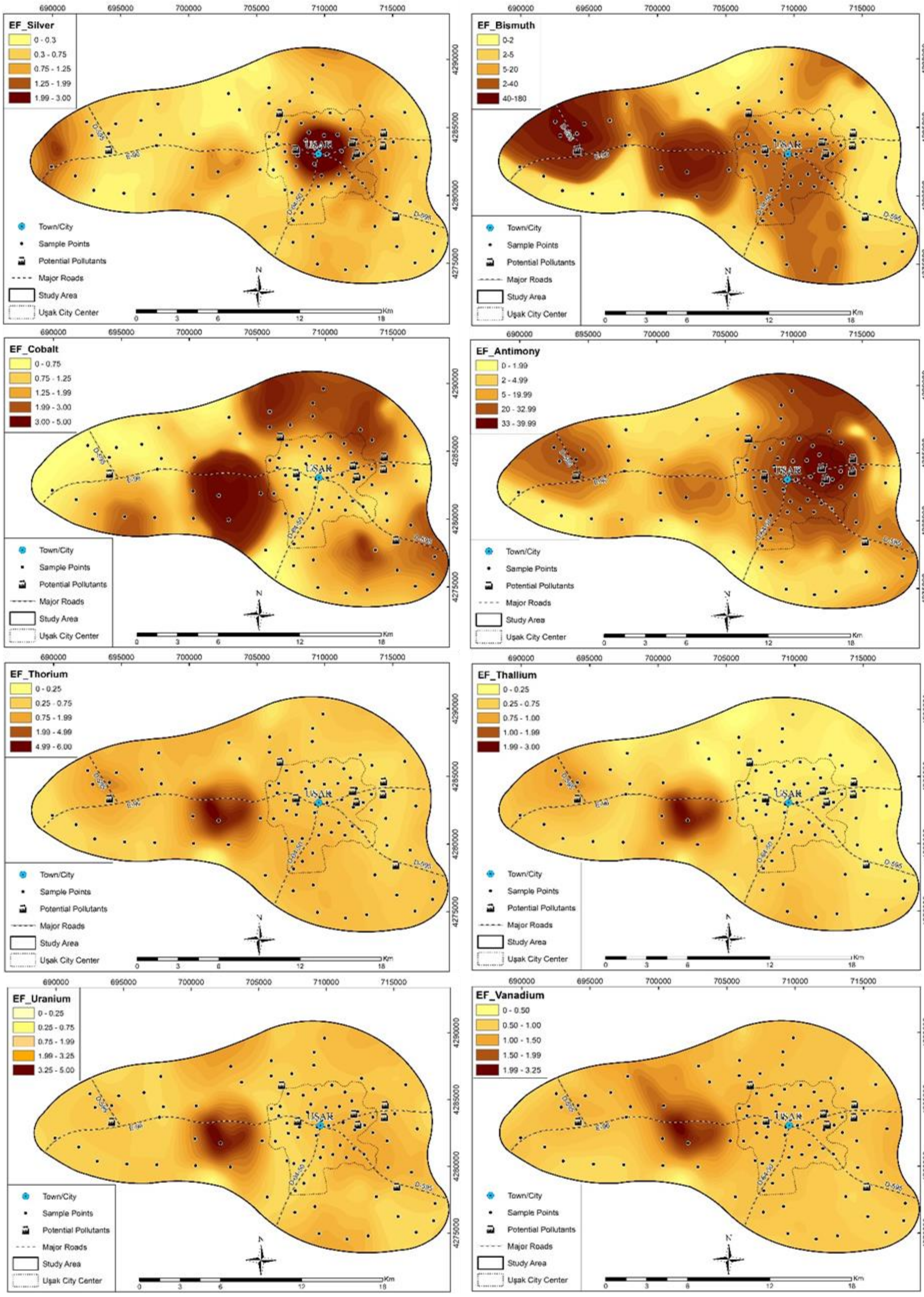

Figure 7. Maps illustrate the variations of the EF values for each heavy metal within the study area.

5. DISCUSSIONS AND CONCLUSIONS

During this research, chemical data from 90 soil samples collected from both urban and agricultural lands of the central province of Uşak was examined to identify ecological pollution and the potential pollutant sources. Igeo and EF indices were utilized to examine the existing ecologic risks both in the urban and agricultural soils of the Uşak. In addition, maps for the heavy metal concentrations, Igeo values, and EF values were generated to observe their spatial distributions within the study area. This study suggests that except for Co, the mean concentration values of Ag, Bi, Sb, Th, Tl, U, and V appear to be in a normal range. Igeo values of Ag showed contamination in the city center that ranges from moderate to heavy, potential pollutant source for the Ag contamination in the city center was interpreted to be due to domestic waste. Igeo values of Bi and Sb in the west part of the study area indicated extremely contaminated soils. These elevated Igeo values appeared to be in the soils around the Uşak Organized Industrial Zone (UOIZ) which might suggest that industrial activities such as paint manufacturing and waste disposal and burning are potential pollutants. Spatial distribution maps of EF values for Bi and Sb also showed significant enrichment in the soils around the UOIZ which further validates that the potential sources for Bi and Sb heavy metals contaminations might be the industrial activities at this specific location.

Finally, the soils of the central province of Uşak have shown pollution associated with the selected uncommon heavy metals. For future research, it is recommended to take more frequent samples by expanding the coverage of the research site. Based on the findings of this study, uncommon heavy metals can also cause soil pollution. It is therefore recommended that uncommon metals should be included in future environmental studies.

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