Research Article

Evaluation of metal contamination and ecological risk in surface sediments of an industrialized catchment: A case study of the Saz-Çayırova catchment, Northwestern Türkiye

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ABSTRACT

Investigating the spatio-temporal variations of metal pollution in the sediment of an industrialized watershed, this study aims to identify ecological risks. Utilizing six risk assessment indices—enrichment factor (EF), geo-accumulation index (I-geo), potential ecological risk (RI), contamination factor (CF), ecological risk assessment (ER), and Pollution Load Index (PLI)—the research distinguishes between anthropogenic and geogenic sources. Surface sediment samples are collected from nine locations (comprising seven monitoring sites and two reference sites) across the watershed during both dry and wet seasons. Reference concentrations, tailored to accurately reflect local characteristics, are employed to compute the indices. Results indicate significantly elevated concentrations of Zn, Pb, Cr, Cd, and Ni throughout the basin, exceeding reference values by factors of 15, 20, 5, 10, and 5, respectively. Wet and dry season assessments reveal varying I-geo and EF values across monitoring stations. Cd emerges as the primary ecological risk, predominantly attributed to industrial discharges. Moreover, dry season contamination surpasses that of the wet season. Comparative analysis of the indices reveals PLI's efficacy for spatial assessments, while RI analysis better elucidates temporal variations. In conclusion, this study provides valuable insights for devising strategies to mitigate sediment contamination in industrial watersheds.

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INTRODUCTION

Heavy metals are regarded essential pollutants for aquatic ecosystems due to their high toxicity, persistence, and biogeochemical accumulation potential [1]. In the last decade, heavy metal pollution has posed a serious environmental threat, particularly with the substantial increase in population and intensive industrialization [2]. Heavy metals reach the aquatic environment through both geogenic and anthropogenic channels, such as geological weathering, soil erosion, intensive transportation, mining, agriculture, and industrial operations that involve hydrolysis and oxidation mechanisms [3, 4]. One of the most important ecological risk of heavy metals is accumulation potential in sediments. The sediments directly affect the quality of water column by decomposing organic matter, leading to the release of macro elements (N, P etc.) and toxic heavy metals, and decreasing the oxygen level of the aquatic system [5]. In this case, the flora and fauna (benthic organisms, carinate, fish, and aquatic plants etc.) are deteriorated irreversibly. Hence, assessment of heavy metal contamination in sediments is critical to determine the pollution level and ecological risks of streams.

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Sediments have frequently been defined as reservoir for a wide variety of pollutants, particularly for heavy metals [6, 7]. In recent studies on sediment quality assessment, the negative impacts of heavy metals on ecology have been evaluated by means of the defined pollution indices [8–10]. In this context, various quality indices including contamination factor (CF), enrichment factor (EF), geo-accumulation index (I-geo) and ecological risk assessment (ER) have been implemented for the assessment. These indices provide advantages such as being simple to use and allowing comparisons across stations with different geographical characteristics, since their background equations are based on normalization. In addition to these advantages, they have disadvantages such as neglecting the reactive properties of heavy metals, not taking into account the changes in heavy metal/reference element ratios due to natural processes, and not being able to determine the weight factor for each heavy metal. Several studies conducted in complex industrialized basins have reported that EF, CF, I-geo indices can give inconsistent results in terms of estimating metal pollution [11–13]. This inconsistency leads to significant uncertainties in the assessment of ecological risk regarding to sediment contamination. Yu et al. [13] have reported that the inconsistency obtained from these indices may be related to the background values and further studies on sediment quality will be needed. Therefore, it is critical to determine the site-specific background values in order to maintain the consistency of the results. Furthermore, in these research, it was suggested to interpret many indices by applying them together rather than relying on a single index.

Several research have utilized recognized global average concentrations, sediment quality criteria, or prior studies performed in or near the examined watershed as reference values [13–15]. Accepting a global reference value on a watershed basis, on the other hand, can lead to high uncertainty due to geogenic, physical, and chemical processes that reflect the local characteristics of the studied watershed [16]. The reference values in sediment quality guidelines, which are based on developed countries, may result in the identification of previously unmonitored industrialized watersheds, as highly dangerous. In such watersheds, the measured values at reference stations, determined through field campaigns based on different soil types and land uses, are essential for assessing the local ecological status of the watershed [8–10].

In Türkiye, various studies have been conducted to determine sediment quality in watershed scale [7, 16–18]. In these studies, the lakes and rivers negatively affected by anthropogenic activities including continuous discharge of artificial pollutants (organic matter and heavy metals) induced by mining, energy generating and complex industrial activities have been reported. These studies focused on a specific pollutant by choosing basins with relatively similar land use and a single branch of industry. In contrast, the watershed area of Saz-Çayırova Stream is a unique aquatic ecosystem in which hydromorphological degradation, complex industrialization, agricultural activities, residential areas, transportation, and abandoned mining activities experienced. This area is ecologically important due to its location within the Türkiye's most important industrial cities of Istanbul and Kocaeli, and the Saz-Çayırova Stream flowing into Sea of Marmara, an inland sea of Türkiye. Any comprehensive sediment metal contamination and ecological risk assessment has not been implemented for the watershed. In the studies conducted on the site, superficial examinations on water quality and land cover changes have only been reported [19–22].

In the light of abovementioned information, this study aims to reveal the temporal (wet and dry periods) and spatial distributions of metal pollution in the sediment of Saz-Çayırova Stream. In this context, ecological risk assessment techniques were used to identify natural and anthropogenic sources of sediment pollution in the watershed. Furthermore, reference values specific to the watershed were calculated based on land use and soil types and compared to other reference values. The outputs of this research are thought to serve decision-makers conduct remediation projects along the stream by identifying metal pollution accumulation regions in the Saz-Çayırova.

MATERIAL AND METHODS

Site Description

The catchment of Saz-Çayırova (40° 45'–40° 52' N, 25° 21'–25° 27' E) is located between the borders of Istanbul and Kocaeli Provinces. The watershed area and average elevation of this catchment are approximately 50 km² and 100 m, respectively (Fig. 1). The drainage area and length of the stream, classified as a small-scale basin, is approximately 20 km² and 10 km, orderly [19]. Residential and industrial areas constitute 73% of the watershed, with individual agricultural lands accounting for 21% and forests accounting for 6% [23]. Furthermore, Saz stream tributaries serve as a receiving environment for the treated wastewater of two Organized Industrial Zones (OIZ-1 and OIZ-2) in the basin's northern region. These regions are composed of approximately 39.7% automotive sub-industry, 20.48% metal industry, 11.7% chemical industry, and 10.4% other manufacturing industries (Fig 1c). The effluents from these industries are rich in heavy metal and nutrient concentrations due to production processes such as dyeing, washing, and phosphating. Other hand, considering that there are 20,000 and 24,000 personnel in OIZ-1 and OIZ-2, respectively, domestic wastewater discharges are also a concern [24].

The elevation in the watershed area varies between sea level and 313 meters. Due to the relatively high elevation difference in the basin, stream energy increases in the downstream parts. In addition, digitized soil maps containing the slope groups and main soil groups are illustrated in Fig 1b and Fig 1d. The watershed is located in the transition zone between the Mediterranean and the Black Sea climate characterized by cold rainy winters, and hot humid summers. The annual average precipitation and temperature are 720 mm, and 15 °C, respectively. The catchment is under prevailing wind direction of NE (70%) [22].
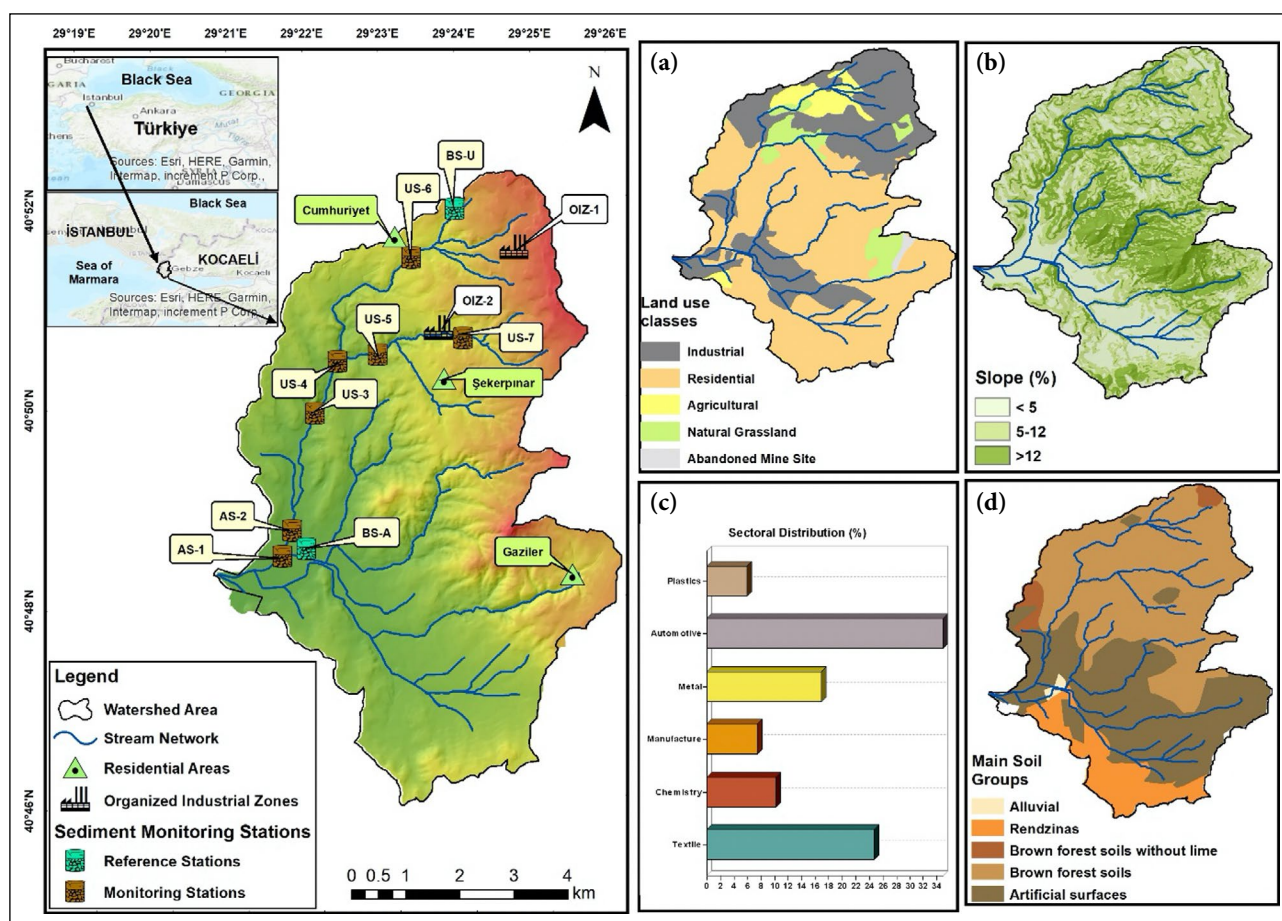
Figure 1. Location map of Saz-Çayırova watershed, **(a)** land use map, **(b)** slope classes, **(c)** sectoral distribution (%) of industries in OIZs, **(d)** main soil groups.

Field Campaigns and Sampling Strategy

The Çayırova stream was rehabilitated by being channelized into concrete after the flood in 2010. Therefore, sediment does not adhere to or accumulate on the concrete floor, especially in the lower part of the Çayırova stream. In this context, sediment quality and potential ecological risk analyzes were carried out only on Saz Stream tributaries. The selection of the sediment sampling stations took into consideration the residential and industrial zones, the stream morphology, and highways. Field campaigns were organized in August and December 2020 to represent dry and wet periods. Surface sediment (0–15 cm) samples were collected at 7 different stations in both periods. Approximately five kilograms of samples were collected with a steel trowel at each sampling station and stored in ten-liter glass jars. Samples were transported to the laboratory in a cooler and then stored in a refrigerator set at 4.0 °C.

The locations of reference stations have been determined by the Instute of Mineral Research and Exploration (MTA) based on digitized geological maps and land use/land cover distribution. Additionally, the number of reference stations has been determined in accordance with the distribution of main soil groups found in the Türkiye National Soil Database (TNSD). The main soil groups of the watershed are distributed as follows (Fig. 1d): Artificial surfaces (35%), brown soils

without lime (37.5%), brown forest soils (11%) and rendzinas (16.5%). The selected monitoring stations along the Saz Stream are situated within regions characterized by brown soils without lime and artificial surfaces. In this regard, two reference stations were selected: BS-U, representing background station for brown forest soils without lime, and BS-A, representing background station for artificial areas. These stations were selected from locations with minimal anthropogenic influence and represent different main soil groups. While the BS-U serves as the reference values for stations between US-3 and US-7, the BS-A was defined as the reference for AS-1 and AS-2 stations. The reference concentrations determined within the scope of this study were compared, with values used at national and international scales.

Laboratory Analysis

The surface sediments collected were first dried in an oven at 105 °C for four hours. After each sample was sieved with a 63 μm diameter sieve, 0.25 grams of sediment sample was burned in a microwave oven (Milestone Ethos 1600) with an acid mixture (HNO_3 – 4 ml, HF-2 ml, $HCIO_4$ -1 ml, H_2O_2–1 ml) in a total volume of 8 ml [25–29]. Afterwards, the samples were diluted to 25 ml with ultrapure water and filtered. All metal analyzes were determined by Inductively Coupled Plasma-Optical Emission Spectrophotometry (ICP-OES, Optima 7000 DV, Perkin Elmer). Preservation,

Table 1. Classification of enrichment factor (EF), geo-accumulation index (I-geo), potential ecological risk (RI), contamination factor (CF), ecological risk assessment (ER) and Pollution Load Index (PLI)

EF scale	Enrichment Level [32]	I-geo classes	Pollution Level [35]
EF<1	No enrichment	I-geo<=0	Unpolluted
EF=1–3	Minimal enrichment	I-geo=0–1	Unpolluted to moderately polluted
EF=3–5	Moderate enrichment	I-geo=1–2	Moderately polluted
EF=5–10	Moderately severe enrichment	I-geo=2–3	Moderately to strongly polluted
EF=25–50	Very severe enrichment	I-geo=3–4	Strongly polluted
EF>50	Extremely severe enrichment	I-geo=4–5	Strongly to very strongly polluted
ER classes	ER Level [38]	CF classes	Contamination Level [37]
ER<40	Low potential ecological risk	CF<1	Low contamination
ER=40–80	Moderate potential ecological risk	CF=1–3	Moderately contaminated
ER=80–160	Significant potential ecological risk	CF=3–6	Considerably contaminated
ER=160–320 & >320	High & Very high potential ecological risk	CF>6	Very high contamination
RI classes	Risk Level [37]	PLI	PLI İndexes [36]
RI<150	Low Ecological risk	PLI<0	Unpolluted soils or sediments
RI=150–300	Moderate ecological risk	PLI=0	Perfection
RI=300–600	Significant ecological risk	PLI=1	Baseline levels of pollutants
RI >600	High ecological risk	PLI>1	Progressive deterioration of the site quality

EF: Enrichment factor; (I-geo): Geo-accumulation index; RI: Potential ecological risk; CF: Contamination factor; ER: Ecological risk assessment; PLI: Pollution load index.

transportation and analysis of sediment samples were carried out according to standard methods [30]. Analytical data quality was ensured by careful standardization, procedural blank samples, and duplicate measurements [31].

Heavy Metal Pollution Indices
In this study, total concentration-based enrichment factor (EF), geo-accumulation index (I-geo), potential ecological risk index (RI), pollution load index (PLI) and ecological risk assessment (ER) indices were used to assess the potential ecological risks of metals in surface sediments. Output of these indices were interpreted according to the following scales (Table 1).

The enrichment factor (EF) is generally used a convenient method to distinguish between geogenic and anthropogenic sources by normalizing chemical components according to their reference values. In many studies, Fe, Al and Ti were recommended as a reference element in EF calculation [32, 33]. The single element normalization approach may cause uncertainty due to the effect of sediment texture changes on the metal concentrations [13, 34]. Furthermore, empirical cumulative density plot and Shapiro-Wilk normality test (alpha <0.05) were applied whole dataset to see outlier values and data distribution, respectively. In our case, Al and Fe was taken as reference element due to do not reveal outlier values and fitted normally distribution. From another perspective, the proximity of the measured concentrations of Al and Fe elements at monitoring stations to those of reference stations supports the suitability of these elements as reference elements. The EF is expressed mathematically [32];

$$EF = \left[\frac{(C/Al)_{sample}}{(C/Al)_{reference}} + \frac{(C/Fe)_{samples}}{(C/Fe)_{reference}}\right]/2 \tag{1}$$

Where C represents the investigated element; Al and Fe are the reference/background elements.

The geo-accumulation Index (I-geo) was proposed by Müller [35] and is widely preferred for evaluating metal/metalloids levels. I-geo was calculated as follows;

$$I_{geo} = \log_2\left(\frac{C_{sample}}{1.5 \times B_{sample}}\right) \tag{2}$$

where C_{sample} is the analyzed concentration (mg/kg) of metal/metalloids in the sample sediment, B_{sample} is the geochemical background value (mg/kg) of the element in the background samples and the factor 1.5 is defined to minimize the effects of possible variations in the background values which may be attributed to lithogenic effects [35]. Measurements from the BS-U and BS-A stations were used to determine reference values for the Saz-Çayırova watershed.

The ecological risk assessment of the watershed was evaluated using the ecological risk index (RI) and the Pollution Load Index (PLI). PLI and RI were calculated as a fraction of the contamination factor (CF) that evaluates the contamination level of a single heavy metal. CF is calculated as follows;

$$CF = \frac{C_{sample}}{C_{reference}} \tag{3}$$

where C_{sample} is the concentration of evaluated metal in the sediment sample and $C_{reference}$ is the background concentration of the heavy metal, specified to the watershed. PLI indexates contamination of metals evaluated for a single monitoring site [36]. It is calculated as a fraction of the CF values;

Table 2. Comparison of reference concentrations (mg/kg) of metals measured with previous studies

Examined elements (mg/kg)	Other studies					This study	
	Coşkun et al. [41]	Yılmaz et al. [42]	Mason [43]	Taylor et al. [46]	Turekian [47]	BS-U	BS-A
Al	–	–	81.300	–	25.000	246.273	88.622
Ti	5.000	–	–	–	1.500	1.512	8.672
Fe	40.000	38.000	50.000	35.000	9.800	68.303	91.134
Ni	50	37	75	75	50	37	100
Cu	30	33	55	55	45	260	327
Zn	90	72	–	–	16	180	729
Cd	BDL	BDL	–	0.2	BDL	BDL	1
Pb	12	37	–	20	20	10	87
Cr	70	61	–	100	90	156	199

*: BDL refers to concentrations below the detection limit.

$$PLI = \sqrt[n]{CF1 \times CF2 \times CF3 \times ..... \times CF_n} \qquad (4)$$

where, n is the number of metals and CF is the contamination factor.

RI is widely used for ecological risk assessment in soil and sediments [37]. The index has been developed for six toxic metals using the following equations [38].

$$RI = \sum_1^n ER \; ; ER = Tr \times CF \qquad (5)$$

where ER is the single index of ecological risk factor, and n is the amount of the heavy metal class, Tr toxic response factor suggested by Hakanson [38] for six metals Ni (5), Cu (5), Zn (1), Cd (10), Pb (5) and Cr (2). ER and RI express the potential ecological risk factor of individual and multiple metals respectively. The expressions and values used for the interpretation of the potential ecological risk factor were reported in Table 1.

RESULTS AND DISCUSSIONS

Assessments of Background Metal Concentrations (mg/kg) in Surface Sediments

Reference concentrations that accurately reflect the local characteristics of a watershed are substantial for the scientific computation of the ecological risk status of that watershed [39, 40]. Because of this, before discussing the results, similarities and differences between the reference values determined within the scope of this research and the reference values commonly used in national and international studies have been discussed (Table 2). While international studies were selected to encompass research on the elements examined in our study, national studies were attempted to be identified among research conducted in regions as close as possible to the Saz-Çayırova.

If we briefly summarize the studies comparing reference values, Coşkun et al. [41] examined the chemical composition of 73 surface soil samples across the Thrace Basin, covering an area of 24,000 km². Additionally, Yılmaz et al. [42] evaluated the chemical composition of 14, 6, and

10 surface soils based on three different land-use classes, namely industrial, urban, and rural areas, in the Izmit Gulf. To compare with the reference values in our study, the average of 30 stations from Yılmaz et al. [42] was calculated and a comparison was made. Mason's [43] study determined the background levels of 23 elements as a consequence of topsoil structure analyses, and these values were used as reference values in numerous subsequent studies on the issue [44, 45]. Taylor et al. [46] also investigated surface soils and contributed to the literature by reporting background concentrations of 22 elements. Turekian [47] analyzed soil samples from 17 distinct locations in the Chattanogour Plateau River Basin, which has a tropical environment, both post- and pre-monsoon. Because of the low anthropogenic influence in the stated area, this study was also used as a reference value in several ecological risk assessments [48, 49].

The Al concentrations observed at BS-A were consistent with Mason's [43] reference values (Table 1). In general, Fe, Ni, Cu, Zn, Cd, Pb, and Cr concentrations measured at the BS-A were found to be higher than in other studies. In particular, Zn, Cu, and Pb appear to be approximately ten times higher than those of the other studies. These high concentrations can be associated with an abandoned metallic sulfide mining site located in the northwest and southeast sectors of the Saz-Çayırova, which has an approximate reserve of 110,000 tons of Pb, Zn, and Cu. In the examined studies, only Taylor et al. [46] measured Cd over the detection limits (0.2 mg/kg), and this amount is around one-fifth of the Cd concentration in our analysis.

Al and Fe levels at the BS-U reference site, which represents brown forest soils without lime, were found to be high relative to other studies. Iron and aluminum oxides tend to accumulate in this type of soil, especially due to humid and hot climatic conditions [50]. In this status, comparatively high reference values for Al and Fe might be considered an expected condition. This study noticed that the Cd concentration was lower than reported in other studies and similar to the value detected by Yılmaz et al. [42]. Yılmaz et al. [42]'s
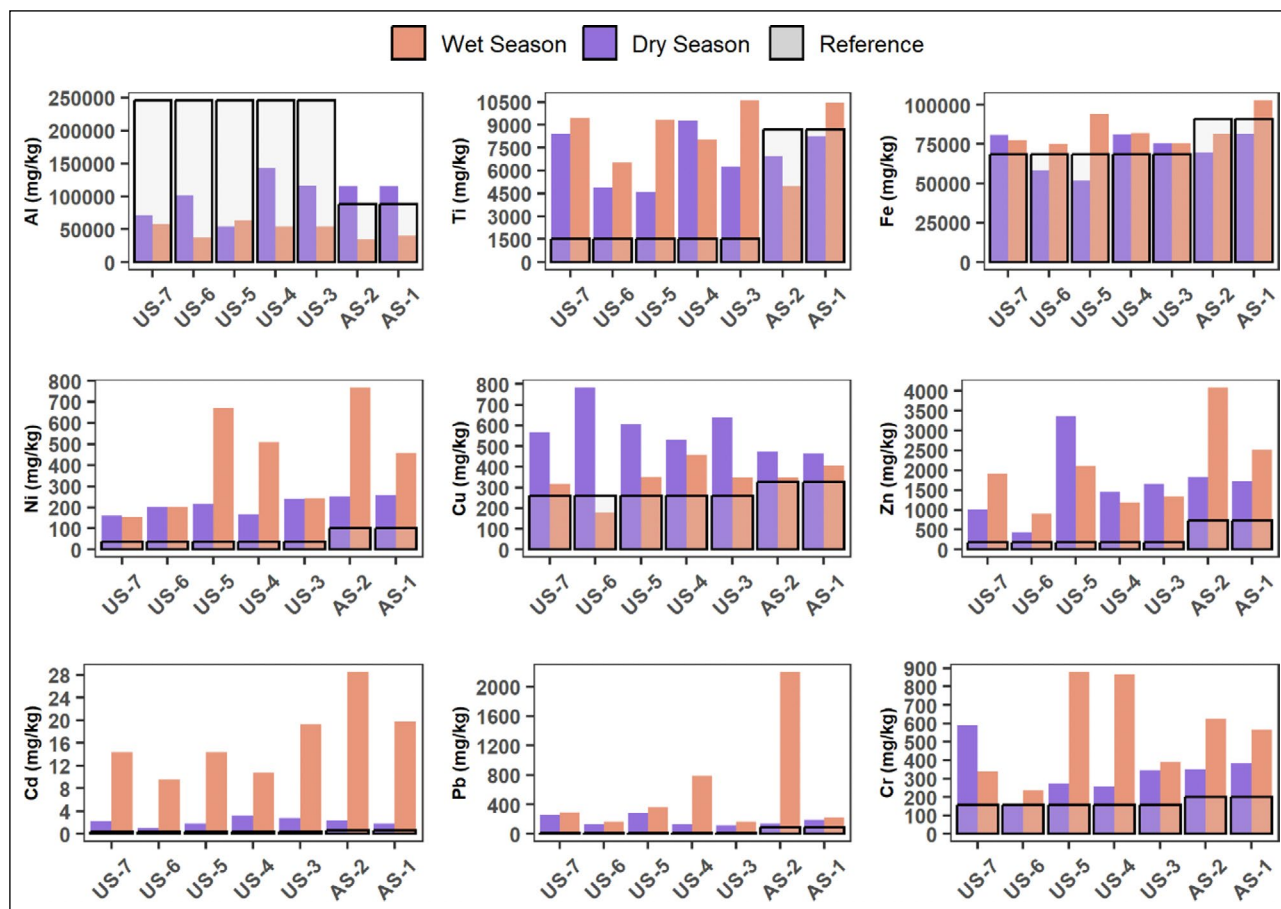
Figure 2. Spatio-temporal variation of metal concentrations throughout the Saz-Çayırova and their comparison with reference concentrations.

research region is close to the Saz-Çayırova basin, which can explain the similarities. The observed Pb was similar to that found by Coşkun et al. [41] and lower than in other studies. Cu, Zn, and Cr concentrations were significantly high relative to the studies compared.

The metal concentrations measured in BS-A and BS-U exhibit both similarities and differences compared to other studies. For instance, despite the proximity of the watershed examined by Yılmaz et al. [42] to Saz-Çayırova, differences were observed in Fe, Al, Cu, and Zn concentrations. This example demonstrates the importance of identifying the local ecological risk dimension of a watershed based on the relatively clean stream network within the studied watershed.

Seasonal and Spatial Variation of Examined Metals

As a preliminary assessment of ecological risk analysis methods, Figure 2 illustrates the seasonal and spatial variations of the examined elements in the watershed, supported by concentrations obtained at reference sites. The concentrations of Al, Ti, and Fe did not differ significantly between the dry and wet seasons, except for some sites. Al concentrations in AS-1 and AS-2, which represent the downstream parts of the watershed, were measured to be high in the wet season. Ti concentrations at US-6 and US-5 were around 1.5 times higher during the dry season than during the wet season.

Ni and Cr were measured to be three times higher in the dry season than in the wet season in the US-4 and US-5. The two stations mentioned represent the outlets of the different stream tributaries (Fig. 1). That's why, there might have been a tendency for the accumulation of nickel and chromium in the surface sediments of the tributary outlets, accompanied by a decrease in the river's energy due to relatively low flow conditions (Fig. 2). Cd and Pb concentrations were significantly higher during the dry season. Pb and Zn levels are roughly 20 and 4 times measured in the dry season relative to wet season, particularly at AS-2 site. There is heavy vehicle and truck traffic approximately 100 meters north of AS-2. It is noted that the main pollutants released into the atmosphere from exhaust emissions are Pb and Zn, and they tend to fall on the earth through atmospheric inversion [51]. In this context, this high difference between the dry and wet season can be associated with vehicle and truck emissions. From another perspective, this can be linked to the transport of metals by surface water, as relatively high flow conditions during the wet season minimize metal accumulation in sediment.

Spatially, except for Zn, the elements investigated for the wet season did not show a high variation from the upstream to the downstream of the watershed. In the dry season, all metals except Al, Ti, Fe and Cu have high variation. It can be said that discharges from individual
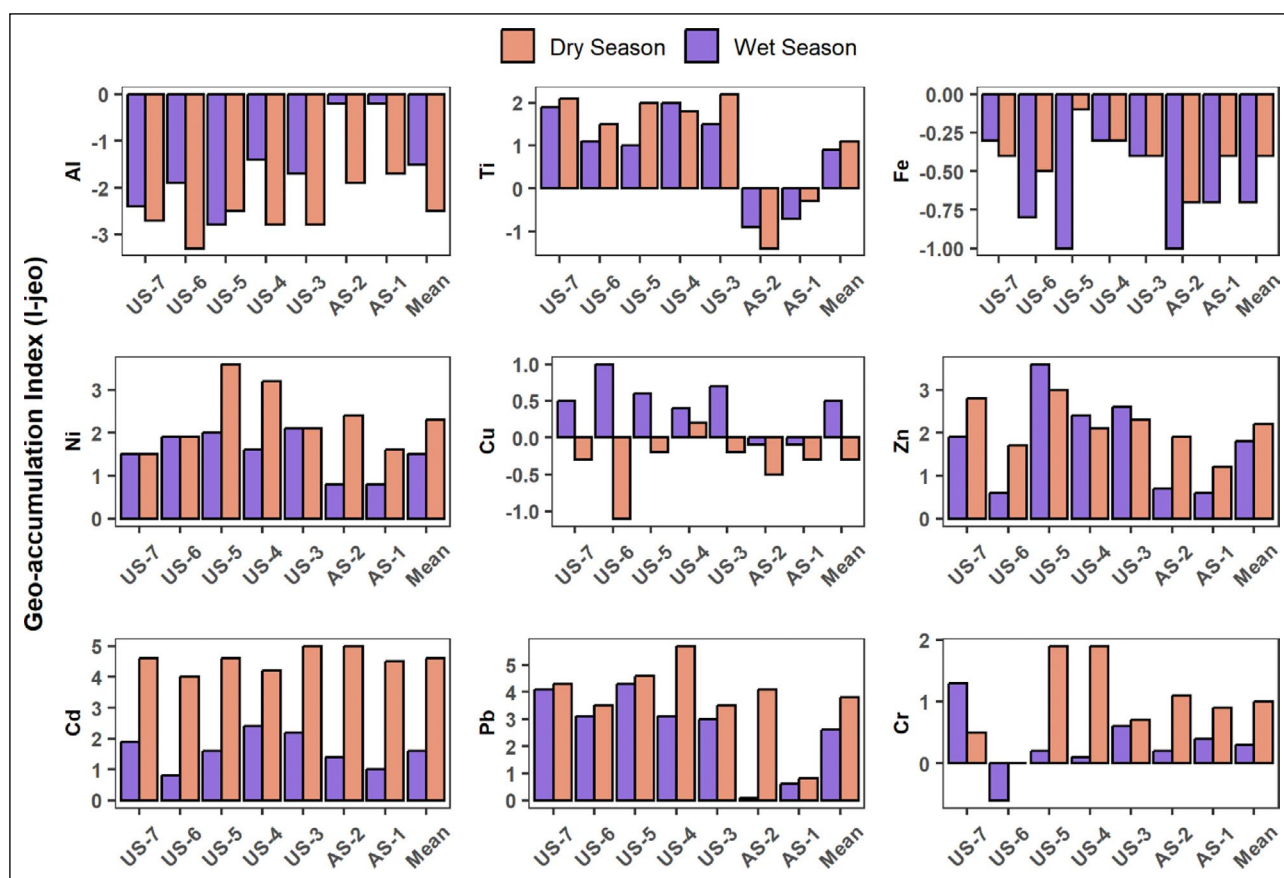
Figure 3. Geo-accumulation (I-geo) values of the metals analyzed in surface sediments.

industries detected by field observations are important factors that trigger the high variation in the dry season. Throughout the watershed, all metals (except Al and Fe) were measured significantly higher than the reference values. Specifically, it was seen that the Zn, Pb, Cr, Cd, and Ni were about 15, 20, 5, 10, and 6 times greater than the reference values, respectively. This clearly illustrate the impact of local activities on surface sediments, especially during the dry season.

Metal Pollution Indices

Enrichment Factor (EF)
EF values were calculated for the wet and dry seasons to identify natural and anthropogenic sources of metal concentrations (Table 3). Al and Fe were excluded from the analysis as they served as reference elements.

During the dry season, all stations showed the extremely severe enrichment Cd enrichment, indicating that this element was the principal contaminant compromising sediment quality. Pb concentrations showed very severe enrichment (US-3) and extremely severe enrichment (US-7, US-6, US-5, US-4, and AS-2). Cr and Ni elements have moderate enrichment in all stations except US-5, US-4 and US-3. That is, very severe enrichments of Pb, Ti, Ni, and Cr were recorded in the two tributaries under industrial pressure (US-4 and US-5), as well as at combination of two tributaries (US-3). US-4, the receiving en-

vironment of OIZ-1, demonstrated the highest Pb and Ni enrichment, implying these tributaries act as metal reservoirs. This localized accumulation necessitates further study of potential impacts and solutions. The Cu was minimal to moderately enriched across the entire watershed while the Zn exhibited very severe enrichment all of sites, except AS-1.

During the wet season, the strongest enrichment was observed in Pb values, and all stations except AS-2 and AS-1 showed very severe enrichment (US-6, US-4, US-3) and extremely severe enrichment (US-7, US-5). The minimal enrichment recognized at the AS-2 and AS-1 sites is directly related to the high Pb concentration (87 mg/kg) reported at the BS-A which represents the reference value for artificial surface. The highest enrichments of Zn were calculated at US-5 (EF = 45.4) and US-7 (EF = 11.8). Given that these sites are situated along the tributaries under the influence of OIZ-2 discharges, it is reasonable to attribute the primary source of zinc in this tributary to OIZ-2 discharges. At the US-5, EF values of the Ti, Ni, and Cd were categorized as extremely severe enrichment. Analyses strongly support that Zn, Ni, Ti, and Cd enrichments originate from OIZ-2 discharges. Because the OIZ-2 includes numerous sectors, such as metal, plastic, textile, and chemical product manufacturing, the wastewater produced by these sectors contains elements such as Zn, Ni, Cd, Ti, As, and Cu [22]. In addition, Cr concentrations had lower EF values than other elements.

Table 3. Enrichment factors (EF) determined in surface sediments throughout the Saz-Çayırova

Sites	Seasons	Ti	Ni	Cu	Zn	Cd	Pb	Cr
US-7	Wet	11.9*	7.8	3.9	11.8*	10.2	65.4**	4.8
	Dry	16.1*	9.2	2.4	23.3*	80.6**	51.4**	6.9
US-6	Wet	5.9	18.8*	4.2	3.2	3.5	17.7*	1.4
	Dry	16.5*	7.5	2.4	17.3*	84.3**	57.3**	5.3
US-5	Wet	10.9*	14.2*	5.6	45.4*	11.1*	69.8**	4.3
	Dry	14.3*	32.8*	2.7	23.6*	74.3 **	74**	11.4*
US-4	Wet	7.9	4.2	1.9	7.6	7.7	12.3*	1.6
	Dry	14.4*	36.7*	4.2	15.6*	65.7**	191.5**	13.3*
US-3	Wet	6.2	7.6	2.9	10.8*	8.4	14.0*	2.6
	Dry	19.3*	15.4*	3.1	17.6*	116.0**	38.8*	15.9*
AS-2	Wet	0.8	2.5	1.5	2.5	4.0	1.6	1.8
	Dry	1.1	16.5*	2.3	12.0*	102.2**	54.1**	6.7
AS-1	Wet	0.9	2.3	1.3	2.1	2.7	2.0	1.7
	Dry	1.9	7.0	1.9	5.2	50.1**	3.8	4.3
Average	Wet	7.1	8.2	3.0	11.9*	6.8	26.1*	2.6
	Dry	11.9*	17.9*	2.7	16.4*	81.9**	66.7**	7.7

*: Represents elements included in the very severe enrichment. **: Represents elements included in the extremely severe enrichment.

In the wet season, average EF values across measurement sites follow the descending order: Pb > Zn > Ni > Cd > Ti > Cu > Cr. In the dry season exhibits a distinct pattern with Cd > Pb > Ni > Zn > Ti > Cr > Cu. Different enriched elements suggest that surface sediment chemistry in the Saz-Çayırova basin is dynamically influenced by seasonal variations. The observed seasonal variations in dominant enriched elements can be attributed to two key factors: (i) increased accumulation of industrial pollutant loads within sediments during low-flow conditions, favoring their retention, and (ii) individual agricultural activities carried out in the US-6 region during the dry season (Fig. 1a).

Geo-Accumulation Index (I-geo)
Geo-accumulation values (I-geo) of the elements examined within the scope of the study were visualized in Figure 3. As expected, Al and Fe exhibited consistently unpolluted levels (I-geo ≤0) throughout the watershed. Other elements demonstrate various pollution level, ranging from Unpolluted to moderately polluted (I-geo=0-1), moderately polluted (I-geo=1-2), moderately to strongly polluted (I-geo=2-3), strongly polluted (I-geo=3-4), and strongly to very strongly polluted (I-geo=4-5). These variations were linked to the combined influence of localized activities within the watershed and natural geochemical processes.

In the dry season, Cd and Pb indicated the highest I-geo values. Cd levels were consistently classified as strongly to very strongly polluted across all sites, with relatively low spatial variation. Pb values represented strongly polluted at all stations except AS-1 (Unpolluted to moderately polluted) and US-3 (strongly polluted). Zn and Ni displayed

the highest I-geo value at the US-5 and the lowest value at the AS-1. Ti indicated moderately to strongly polluted level in the Saz tributaries. However, with the addition of the Çayırova tributaries, Ti clustered to the unpolluted class. This observation is directly related to the relatively high Ti concentration measured in BS-A. The Cr caused moderately polluted at the US-4 and US-5, while clustering unpolluted levels at other stations. The negative I-geo values observed for the Cu element throughout the watershed (except US-4) point out the absence of sediment contamination and suggest that the Cu found in the sediment originates primarily from natural processes such as soil or rock weathering.

During the wet season, Pb and Zn demonstrated the highest I-geo values, followed by Cd and Ni. The high contamination originating from these elements was calculated in the US-5 and US-7. Considering that these tributaries were influenced by OIZ-2 discharges. Hence, the primary source of Pb, Zn, Cd, and Ni contamination in surface sediments can be attributed to OIZ-2 discharges. The highest and lowest I-geo values for Ti were recorded at US-4 and AS-2, respectively. This illustrates that Ti-rich surface sediments originate from the northern stream tributary. The I-geo values of Cu and Cr indicate unpolluted to moderately polluted throughout the basin, reaching their maximum at the US-7 station. The US-7 station represents the region before the OIZ-2 discharges (Fig. 1). The relatively high I-geo values observed during the wet season can be attributed to erosion.

The average I-geo values of the monitoring stations in wet and dry seasons ranked in descending order are; Pb > Zn > Ni > Cd > Ti > Cu > Cr > Fe > Al in the wet season and Cd

Table 4. Contamination factor (CF) and Pollution Load Index (PLI) values of the Saz-Çayırova

Stations	Seasons	Contamination factor (CF)									PLI
		Al	Ti	Fe	Ni	Cu	Zn	Cd	Pb	Cr	
US-7	Wet	0.3	5.6*	1.2	4.3*	2.2	5.6*	5.6*	26**	3.8*	3.4
	Dry	0.2	6.3**	1.1	4.2*	1.2	11**	37**	29**	2.2	3.9
US-6	Wet	0.4	3.3*	0.8	5.5*	3.0	2.4	2.5	13**	1.0	2.2
	Dry	0.1	4.3*	1.1	5.4*	0.7	5.0*	24**	17**	1.5	2.7
US-5	Wet	0.2	3.0	0.8	5.8*	2.3	19**	4.6*	29**	1.8	3.1
	Dry	0.3	6.2**	1.4	18**	1.3	12**	37**	37**	5.6*	5.5
US-4	Wet	0.6	6.1**	1.2	4.5	2.0	8**	8.1**	13**	1.7	3.4
	Dry	0.2	5.3*	1.2	14**	1.8	6.5**	28**	80**	5.6*	5.2
US-3	Wet	0.5	4.1*	1.1	6.5**	2.5	9.1**	7.1**	12**	2.2	3.4
	Dry	0.2	7.0**	1.1	6.5**	1.3	7.5**	49**	17**	2.5	3.9
AS-2	Wet	1.3	0.8	0.8	2.5	1.4	2.5	4.0*	1.7	1.8	1.6
	Dry	0.4	0.6	0.9	7.7**	1.1	5.6	48**	25**	3.1*	3.2
AS-1	Wet	1.3	1.0	0.9	2.6	1.4	2.3	2.9	2.2	1.9	1.7
	Dry	0.5	1.2	1.1	4.6*	1.2	3.5	33**	2.5	2.8	2.4
Average	Wet	0.7	3.4*	1.0	4.5*	2.1	7.0**	5.0*	14**	2.0	2.7
	Dry	0.3	4.4*	1.1	8.6**	1.2	7.2**	36**	30**	3.3*	3.8

*: Represents elements included in the considerably contaminated class (CF=3–6). **: Represents elements included in the very high contamination class (CF>6).

> Pb > Ni > Zn > Ti > Cr > Cu > Fe > Al in the dry season. The highest I-geo values in both seasons can be explained by the reasons mentioned in the EF analysis. Furthermore, a high consistency is observed between I-geo and the EF analysis results. This strong consistency indicates that metal pollution indices serve as a significant reference for evaluating ecological risk assessment.

Ecological Risk Assessment Indices

Contamination Factor (CF) and Ecological Risk Assessment (ER)
Contamination factor (CF) values were found to be similar to those of EF and I-geo analyses (Table 4). This can be attributed to the fact that all three analyses rely on similar mathematical equations, despite employing different normalization techniques. The mean Pb and Zn values indicate a very high contamination level in both wet and dry seasons, while Cd and Ni included this group in the dry season. The mean Cr values obtained for the dry season and Ti also emerge as considerable pollutants across the watershed.

Ecological Risk Assessment (ER) results refer to lower potential risk classes (low-medium-significant) against the previous three analyzes (EF, I-geo, CF). For instance, although Pb was the element with the high enrichment and contamination throughout the watershed, it was clustered in the low, moderate, and significant potential ecological risk levels in ER values, except for the US-5 and US-4 (Table 5). A similar situation is valid for Cu, Zn, and Cr elements. In contrast, Cd poses a high and

very high potential ecological risk in the dry season for all sites, as calculated in EF, I-geo, and CF techniques. This is related to the toxic response factor (Tr) determined for each heavy metal in the ER calculation. The high Tr value of 10 assigned to Cd, when multiplied by the contamination factor values, reveals Cd as the most critical parameter ecologically for the Saz-Çayırova. The main pollution sources of Cd are metal industries, metal mining, and the refining, production, and application of phosphate fertilizers [52, 53]. Metal industry activities within the watershed seem to affect Cd concentrations more than other heavy metals.

Seasonal and Spatial Variation of PLI and RI Values
The spatial variability of PLI across the Saz stream indicates progressive deterioration of the site quality (PLI>1) during both dry and wet seasons (Fig. 4). In the wet season, PLI peaked at stations US-7, US-4, and US-3, while the lowest values were found in downstream parts of the watershed. During the dry season, the maximum PLI (5.5) was recorded at US-5, while the lowest (2.4) was recorded at AS-1. The average PLI of the wet and dry periods highlights increased pollution in the dry season. Notably, the stations exhibiting maximal Pollution Load Index (PLI) values differ between wet and dry seasons. Besides, all share the key feature of being located on drainage networks impacted by OIZs. This suggests that surface sediments in OIZ-2 influenced stream networks during the dry season and those under OIZ-1 pressure during the wet season pose potential ecological threats.

Table 5. Ecological Risk Assessment (ER) and Potential Ecological Risk Index (RI) values in surface sediments

Stations	Seasons	Ecological risk (ER)					RI
		Cu	Zn	Cd	Pb	Cr	
US-7	Wet	10.9	5.6	167.9*	131.1	7.6	323.0
	Dry	6.1	10.6	1095.5**	148.0	4.3	1264.5
US-6	Wet	15.1	2.4	76.2	64.7	2.0	160.2
	Dry	3.4	5.0	727.4**	82.4	3.1	821.2
US-5	Wet	11.6	18.7	137.2	143.8	3.5	314.9
	Dry	6.7	11.7	1100.9**	182.8*	11.3	1313.4
US-4	Wet	10.2	8.1	244.3*	65.2	3.3	331.1
	Dry	8.8	6.5	825.9**	401.2**	11.1	1253.6
US-3	Wet	12.3	9.1	213.4	59.7	4.5	289.0
	Dry	6.6	7.5	1475.9**	82.3	5.0	1577.2
AS-2	Wet	7.2	2.5	119.8	7.9	3.5	140.9
	Dry	5.3	5.6	1426.6**	126.0	6.3	1569.7
AS-1	Wet	7.1	2.3	88.5	11.0	3.8	112.8
	Dry	6.2	3.5	989.6**	12.6	5.7	1017.5
Average	Wet	10.6	7.0	149.6	69.1	4.0	240.3
	Dry	6.2	7.2	1091.7**	147.9	6.7	1259.6

*: Represents high potential ecological risk level (ER=160–320). **: Represents very the high potential ecological risk level (ER=160–320).

The spatial distribution of Potential Ecological Risk Index (RI) values varies during the wet season (Fig. 4). While AS-1 and AS-2 stations are classified as low ecological risk (RI<150) during the wet season, US-3 and US-6 are classified as moderate ecological risk (RI=150-300). US-7, US-5 and US-4 stand out as significant ecological risk (RI = 300-600). The average RI value for the wet season (RI = 240.3) showed that surface sediments pose a moderate ecological risk. US-5 and US-4 representing outlets of the tributaries before joining the main network, explain their high RI values compared to others. As discussed previously, relatively high background levels at AS-1 and AS-2 contributed to their low RI values.

In the dry season, the distribution of RI values throughout the watershed was similar to PLI values. Across all monitored stations and the average value of RI (1259.6), surface sediments pose a high ecological risk. AS-2 (RI=1569.7) and US-3 (RI=1577.2) carried the distinction of being the highest-risk sites, while US-6 (RI=821.2) was the lowest (Table 4). The Tr values determined for Cd at all stations are the basis for the high RI values. Cd tends to accumulate in surface sediments in the dry season. Many studies have linked the element Cd to colloidal particulates that can be easily transported via surface flow in streams [54, 55]. Essentially, Cd carried by these readily transported colloids, primarily sourced from local industries, generates significantly elevated ecological risk under low-flow conditions.

While PLI and RI values agree in the dry season, a significant difference occurs during the wet season. Both PLI and RI results were impacted by Cd, but RI reveals this influence more distinctly due to fewer included elements. Excluding Cd reclassifies almost all stations (except US-4 in the dry season) as low-risk in RI, highlighting its sensitivity to dominant pollutants. Conversely, eliminating Cd had low effect on PLI's classification of progressive deterioration of the site quality demonstrating its weak ability to identify specific dominant variables. As demonstrated by this example, RI analysis allows for the determination of the dominant pollutant(s) in surface sediments, as well as a more direct effect of the dominant pollutant(s) on the results of this type of analysis. The limitation of RI analysis is that it can only test a total of eight heavy metals, including Cu, Zn, Cd, Pb, and Cr, all of which were investigated in this study.

CONCLUSION

The ever-expanding presence of urbanization and industrialization in watersheds amplifies the current sediment's sensitivity to environmental changes. Frequent activities like recreation, construction, and reclamation directly impact sediment characteristics, posing a risk to the delicate ecological balance. To effectively manage these impacts, high-resolution spatial and temporal monitoring of recipient environment components is crucial. Such data forms the vital foundation for informed pollution prevention strategies. As proven by this research, monitoring and char-
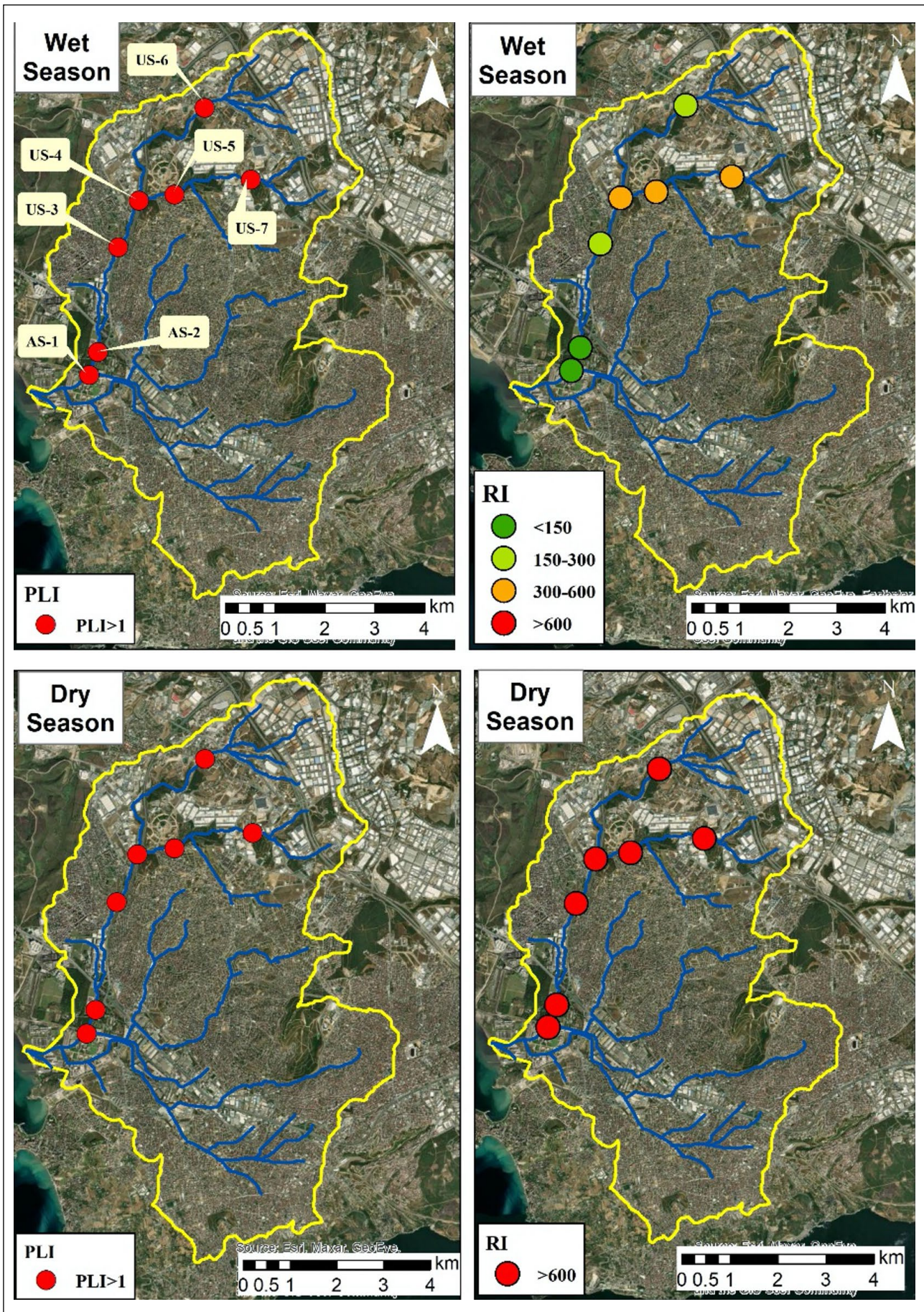
Figure 4. Spatial distribution of PLI and RI values of surface sediments collected during the dry and wet seasons.

acterization of sediment quality are essential for interpreting and quantifying the dynamic responses of these watersheds, ultimately aiding sustainable management practices.

This research evaluates the seasonal pollution levels of surface sediments in the Saz-Çayırova using various indices. The obtained results demonstrate variations depending on seasonality and the type of index employed. The RI was more consistent with EF and I-geo, revealing higher contamination levels during the dry season. The PLI analysis yielded more consistent results with EF and I-geo when evaluated on a station-by-station basis. The ranking of stations based on PLI values for the wet and dry seasons was US-4=US-7=US-3>US-6>AS-1>AS-2 and US-5>US-4>US-3=US-7>AS-2>US-6>AS-1, respectively. The RI values followed the order US-4>US-7>US-5>US-3>US-6>AS-2>AS-1 and US-3>AS-2>US-5>US-7>US-4>AS-1>US-6 for the wet and dry seasons, respectively. The EF and I-geo analyses showed comparable tendencies to the PLI analysis in both seasons. These finding suggest that RI analysis is more trustworthy for assessing temporal variations in the ecological risk of surface sediments, but PLI analysis gives a more consistent evaluation of spatial variations. The improved consistency of PLI in assessing spatial variations can be attributed to the inclusion of elements used in metal pollution indices like EF and I-geo. The better consistency of RI analysis in capturing seasonal differences stems from the flexible range of its scale compared to PLI analysis.

Future research should focus on modeling transport processes in addition to point-based monitoring of the sediment chemical composition. Studies incorporating computational approaches, particularly those addressing surface water-sediment and groundwater-sediment interactions, will provide crucial information for ecological risk assessment to decision-makers.

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DATA AVAILABILITY STATEMENT

The author confirm that the data that supports the findings of this study are available within the article. Raw data that support the finding of this study are available from the corresponding author, upon reasonable request.

CONFLICT OF INTEREST

The author declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

USE OF AI FOR WRITING ASSISTANCE

Not declared.

ETHICS

There are no ethical issues with the publication of this manuscript.

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