



Investigation of Heavy Metal Pollution in the Sediments of the Karasu Stream, Sinop Province

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Research Article

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Abstract

The purpose of this study was to determine possible causes of contamination and assess the sediment quality of the Karasu Stream in Sinop Province. Between April and September 2023, sediment samples were collected from three distinct locations and their heavy metal concentrations was examined. Results show that human activities, especially pollution sources from residential areas, have a major impact on the stream's water quality. Station 2, which is located near densely populated areas, had the greatest quantities of nutrients, organic contaminants, and heavy metals. On the other hand, Station 1, which is close to the stream's source and far from human habitation, had the lowest amounts, most likely as a result of the natural environment. While Ni indicated considerable pollution and enrichment at several sites, overall heavy metal contamination was modest and did not pose an ecological risk, according to pollution indices (Cf, Cd, mCd, EF, and Igeo). The study's findings highlight the necessity of taking the required actions to preserve and enhance Karasu Stream's water quality.

Keywords: Sediment quality, trace elements, pollution assessment, Karasu Stream, Sinop

Sinop İli Karasu Deresi Sedimentlerinde Ağır Metal Kirliliğinin İncelenmesi

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Öz

Bu çalışmanın amacı, Sinop ilindeki Karasu Deresi'nin olası kirlenme nedenlerini belirlemek ve sediman kalitesini değerlendirmektir. 2023 yılının Nisan ve Eylül ayları arasında, üç farklı noktadan sediman örnekleri toplandı ve bunların ağır metal konsantrasyonları incelendi. Sonuçlar, insan faaliyetlerinin, özellikle yerleşim alanlarından kaynaklanan kirlilik kaynaklarının, derenin su kalitesi üzerinde önemli bir etkiye sahip olduğunu göstermektedir. Nüfus yoğunluğu yüksek alanların yakınında bulunan İstasyon 2, en yüksek miktarda besin maddesi, organik kirlileti ve ağır metal içerirken, derenin kaynağına yakın ve insan yerleşimlerinden uzak olan İstasyon 1, muhtemelen doğal ortamın bir sonucu olarak en düşük miktarlara sahipti. Birkaç bölgede önemli ölçüde kirlilik ve zenginleşme olduğunu gösterirken, kirlilik endekslerine (Cf, Cd, mCd, EF ve Igeo) göre genel olarak ağır metal kirliliği düşük düzeydeydi ve ekolojik bir tehlike oluşturmuyordu. Çalışmanın bulguları, Karasu Deresi'nin su kalitesini korumak ve iyileştirmek için gerekli önlemlerin alınması gerektiğini vurgulamaktadır.

Introduction

Approximately 75% of the Earth's surface is covered by salt water. In contrast freshwater resources including rivers, lakes, groundwater, and polar ice caps constitute about 3% of the total water on Earth. Of this limited freshwater supply, only 0.27% is found in rivers and lakes, which are directly accessible to living organisms for use [1]. Among the critical challenges of the 20th and 21st centuries is environmental pollution. Environmental pollution is increasing due to rapid urbanization, industrial activities and population growth rate [2]. The ecosystems that are most affected by environmental pollution are primarily rivers and lakes. Industrial and domestic wastes mixed into water resources directly or indirectly impair water basins. Heavy metals cause inorganic pollution in aquatic systems. Heavy metals that pass into water through various means threaten the metabolism of living beings [3]. With the rapid advancement of industrialization, the world is faced with numerous threats, water pollution being one of these dangers. Water pollution is defined as the deterioration of water quality to the extent that water resources cannot be used as a result of radioactive, biological, inorganic and organic substances mixing into water resources at a level that will damage or prevent their use [4, 5]. According to Ustaoglu [6], water pollution is generally explained as the adverse changes in the biological, chemical or physical properties of water caused by human activities, but it has also been explained by various definitions. For example; FAO defines water pollution as "the discharge of substances into the water that are harmful to living resources, dangerous to human health, hinder activities such as fishing, and may damage water quality" [6, 7]. Pollution in water is addressed under three headings chemical, biological and physical—depending on the pollutant type. It has been reported that water is rapidly becoming polluted beyond recycling due to reasons such as sewage waste, mining operations, food industry waste, agricultural waste, industrial waste, natural disasters and industry. Transition elements, commonly known as heavy metals, are significant contributors to chemical pollution. Heavy metals defined by an atomic number above 20 and a density exceeding 5 g/cm³ are known for their toxic effects and their role in environmental pollution. There are more than 60 metals in this group, including metals such as Zn, Cu, Cd, Pb, Fe, Co, Cr, Hg, Ni [8-11]. From a biological perspective, heavy metals are defined as pollutants that cause environmental pollution and exhibit high toxicity [12]. Heavy metals enter the environment through wind-borne dust, rock fragments dislodged by erosion, volcanic activity, factory waste, domestic waste, forest fires, sewage, and contaminated water sources, such as lakes and rivers, and affect the metabolism of living organisms in various polluting ways [3, 5]. Heavy metals, which are constantly carried by water, dilute greatly over time and some of them become solid as sulfate, sulfide or carbonate components, which settle at the water bottom and cause the sediments in the region to become enriched in heavy metals. Since the absorption capacity of the sediment layer is limited, the heavy metal concentration of the water gradually increases [13]. Therefore, sediment analysis plays a

major role in determining pollution levels in aquatic environments. The study examined heavy metal (As, Pb, Cd, Cr, Fe, Mn, Cu, Co, Ni, Zn) pollution in Bangladeshi rivers between 2001 and 2020 and found that levels significantly increased between 2011 and 2020 compared to the previous decade. In particular, the threshold values for As, Pb, Cd, Cr, Fe, and Mn were exceeded in most regions, with the Buriganga River in Dhaka emerging as the most polluted area. The primary sources of pollution were identified as industrial, agricultural, and mining activities, highlighting serious risks to public health and ecosystems [14]. Wang and Chadwick's meta-analysis examined the accumulation of Pb, Cu, Zn, Cd, Cr, Ni, As, and Hg in China's nine major river systems revealing that Cd, Cr, and Hg levels in river water in the Huaihe and Haihe systems exceeded standards by 20–40 times, and that Hg, Cd, Cu, and Zn accumulation in sediments was particularly high in the Songliao, Yellow River, and Southeast Drainage regions. The study emphasised that Cr in the Huaihe River poses a carcinogenic risk to human health, particularly through ingestion and skin contact, while Cd and Cu pose a high ecological risk in sediments. It also found strong correlations between metals, concluding that cumulative pollution could create more serious environmental and health threats [15]. The mobility, bioavailability and effects of heavy metals in river sediments on ecosystems were examined and it was found that environmental factors such as pH, redox conditions, sulphur and salinity significantly affected this process. The study emphasises the need to consider organic carbon as a critical component in long-term sediment management [16]. In research conducted in a transboundary basin covering the Bangladesh-India border, the source distribution, ecological risks and controlling factors of heavy metals in river sediments were evaluated based on a receptor model. The findings revealed that industrial and agricultural activities are the primary sources of pollution, that some metals pose high ecological risks, and that factors such as pH, particle size, and organic matter content significantly influence metal distribution. In conclusion, it was emphasised that the risks posed by heavy metals in sediments must be carefully monitored for ecosystem health and regional management [17]. In a study conducted in the Dianchi Basin, China, heavy metal pollution, sources and risks in the sediments of the river-lake system were investigated based on empirical evidence. Analyses showed that Cd, As and Pb in particular exceeded the limit values and exhibited high pollution levels. Source estimates indicated that these metals primarily originated from industrial activities, mining, and agricultural inputs, while ecological risk assessments highlighted serious hazards in some areas. The study emphasised that controlling heavy metal inputs is essential for the sustainable management of the basin ecosystem [18]. Evidence from the Yangtze River Basin indicates that the health risks associated with sources of heavy metals in surface water were assessed using probabilistic methods. The findings showed that As, Cr, and Pb were released into the water, particularly from industrial and agricultural sources, and posed risks above acceptable limits for drinking water use in some areas. The analyses revealed that children were exposed to higher health risks compared to adults, while total carcinogenic and non-carcinogenic risks were found to vary depending on the source. The study highlights the importance of sector-specific control and prevention measures

to reduce heavy metal pollution in the river basin [19]. Within the context of research carried out in the estuary region of the Dagu River, heavy metal accumulation in surface sediments and potential risks were evaluated. The results showed that Cd and Pb accumulated significantly and were prominent in terms of ecological risk. The order of accumulation was determined as $As > Cd > Cu > Pb > Zn > Cr$. Risk analyses revealed that heavy metals in some sampling areas could pose a moderate to high risk to aquatic life, while source assessments showed that pollution was mainly caused by industrial discharges and agricultural activities. The study emphasised the need for continuous monitoring and control of heavy metal pollution to protect estuary ecosystems [20]. As observed in studies of an urban river in Turkey, the effects of domestic and industrial wastewater treatment plants on microplastic pollution were evaluated. The findings revealed that the concentration of microplastics in the treatment plant effluents was high and that these discharges significantly increased the total microplastic load in the river. It was determined that microplastics were mostly in the form of fibres and fragments, with polyethylene and polypropylene being the dominant polymer types. The study emphasised that current wastewater treatment processes cannot fully prevent the transfer of microplastics into aquatic ecosystems and therefore highlights the necessity of implementing more advanced treatment technologies [21]. In this study conducted in the Pülümür River in Turkey, heavy metal pollution was assessed in surface water and sediment samples. Analyses showed that sediments contained particularly high concentrations of Cr, Ni and Pb, while some metals in surface water periodically exceeded standard limits. Ecological risk assessments revealed that Cr and Ni in sediments could pose a moderate to high risk to aquatic life. The findings emphasised that the natural geological structure of the region and human activities jointly contribute to increased heavy metal loads, and that regular monitoring and control measures are essential for maintaining water quality [22]. Değirmendere River Basin and its estuary region served as the study area, and toxic metal pollution in sediments was examined seasonally and ecological risk levels were assessed. The findings showed that Cd and Pb in particular reached high concentrations in some seasons and posed serious ecological risks. Additionally, it was determined that metal distributions exhibit seasonal variations and that rainfall and flow conditions influence these differences. The study emphasizes that heavy metal contamination in the Değirmendere ecosystem is closely linked to seasonal dynamics and that developing an effective monitoring strategy is of critical importance for reducing ecological risks [23]. This study aims to investigate the presence of heavy metals including chromium (Cr), cobalt (Co), nickel (Ni), copper (Cu), zinc (Zn), cadmium (Cd), aluminum (Al), arsenic (As), lead (Pb), and uranium (U) in the sediments of the Karasu Stream in Sinop province, and to identify the regions most affected by pollution. This study provides an up-to-date dataset on the heavy metal content of sediments in the Kara Su Stream, based on six months of monitoring. The findings contribute to the assessment of regional environmental quality by revealing the current status of the basin. Due to the six-month study period, seasonal variations were only partially observed. Future studies are planned to implement longer-term monitoring programs that cover all

seasons of the year. It is anticipated that this approach will enhance the generalizability of the results by providing a clearer understanding of seasonal changes.

Materials and Methods

Determination of Stations and Collection of Samples

The Karasu Stream, originating from Gündüzlü Forests of the Küre Mountains in the Black Sea Region, flows from south to north and extends to Erfelek district and its waters flow into the Black Sea through the Akliman coast, 8 km west of Sinop province. Karasu Stream is approximately 80 kilometers long and flows in a wide valley. The main streams feeding Karasu Stream are Tasnak, Kınık, Ramlı, Hasan, Çatak and Çakçak streams [24]. Three stations with different characteristics were identified on the Karasu stream. In order to detect environmental pollutants, sediment samples taken from the stream were collected from specific stations based on varying chemical and physical characteristics. Station 1: Çobanlar village, located 16 km away from the Sinop center (coordinates: 36T 667805 4648395). This station is in a region close to the forested area and where agricultural activities are intense. The possibility of chemicals used in agriculture to reach the Karasu Stream through irrigation channels and rainwater was taken into consideration. Station 2: Sinop Airport area (coordinates: 36T 670450 4653100) is located near residential areas, close to the central villages of Sinop where human activities are intense. The region is in the area where agricultural lands are dense and hospital waste is likely to enter the Karasu stream. Station 3: Dibekli Village, Akliman location (coordinates: 36T 670520 4655453). This station represents the area where the Karasu stream meets the sea. It is located within the Akliman settlement area in Sinop city center and is under the influence of the region.

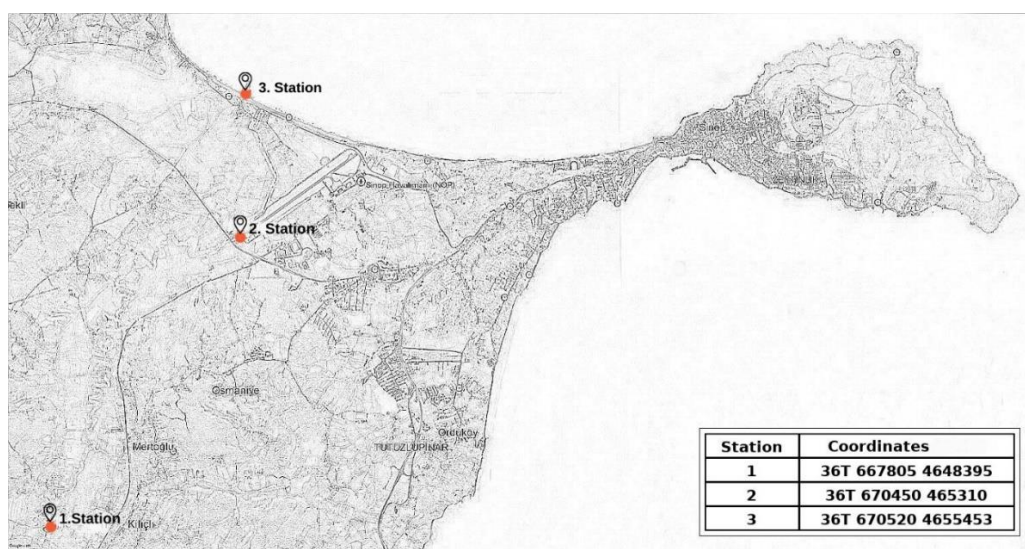


Figure 1. Topographic structure of Karasu Stream and stations

Figure 1 shows the topography of the Karasu Stream along with the sampling stations.

Characteristics of Karasu Stream

The area through which Karasu Stream flows has a temperate Black Sea climate. This climate type is characterized by warm and rainy winters and hot and dry summers. The average temperature in the April-September 2023 period is around 21°C and the average precipitation is 54 kg. In the summer months, especially in August, the amount of precipitation decreases and temperatures increase, causing the stream flow to decrease. The Karasu Stream is fed by both the precipitation regime and groundwater resources in the region. Based on data provided by the General Directorate of Meteorology under the Ministry of Environment, Urbanization and Climate Change of the Republic of Türkiye, the average precipitation and temperature values for Sinop province during the April–September 2023 period are presented in Tables 1 and 2.

Table 1. Rainfall averages of Sinop Province in the period April-September 2023

Total Monthly Rainfall (mm=kg÷m²) OMGI												
Year/Month	1	2	3	4	5	6	7	8	9	10	11	12
2023				51.8	45.2	140.6	72.8	1.4	12.2			

Table 2. Temperature averages of Sinop Province in the period April-September 2023

Monthly Average Temperature (°C)												
Year/Month	1	2	3	4	5	6	7	8	9	10	11	12
2023				13.0	15.5	21.6	25.3	22.9				

The flow regime of Karasu Stream is controlled by the precipitation regime and groundwater resources in the region. Stream flow is generally high in autumn and winter and low in spring and summer. These seasonal changes influence parameters such as water flow rate, sediment transport, and water temperature in the stream.

Devices Used in the Experiment

Chromium (Cr), cobalt (Co), nickel (Ni), copper (Cu), zinc (Zn), cadmium (Cd), aluminum (Al), arsenic (As), lead (Pb) and uranium (U) concentrations in sediment samples were analyzed using an ICP_MS device at the Scientific and Technological Research Application and Research Center of Sinop University.

Table 3. ICP - MS LOD and LOQ Values

Component	LOD	LOQ
Li	0.3125	0.9375
Be	0.0047	0.0141
B	0.0578	0.1734
Na	0.3825	1.1475
Mg	0.0876	0.2628
Al	0.2546	0.7638
Si	1.3545	4.0635
P	1.2536	3.7608
K	4.2	12.6
Ca	5	15
Ti	0.5432	1.6296
V	0.0232	0.0696
Cr	0.0652	0.1956
Mn	0.1203	3.3609
Fe	0.4664	1.3922
Co	0.0042	0.0127
Ni	0.0366	0.1099
Cu	0.0936	0.2808
Zn	0.9797	2.9391
Ga	0.0713	0.2139
As	0.0069	0.0208
Se	0.4097	1.2291
Rb	0.0034	0.0102
Sr	0.0102	0.0307
Mo	0.0345	0.1036
Ag	0.0036	0.0108
Cd	0.0076	0.0228
Sb	0.0043	0.0129
Cs	0.0078	0.0234
Ba	0.0033	0.0099
Tl	0.0034	0.0102
Pb	0.0049	0.0147
Hg	0.003	0.009
Th	0.0084	0.0073
U	0.0083	0.0032

Standardisation and Quality Control in ICP-MS Analyses

ICP-MS measurements were performed on an Agilent 7700x ICP-MS instrument based on the EPA 200.8 method. During the measurements, 10 mL of each water sample was acidified with 65% HNO₃ (Suprapur) and diluted as necessary to keep the total dissolved solids (TDS) below 0.1%. Multi-element standard solutions provided by Agilent (27-element mix: 8500-6940 2A and 8500-6940 Hg) were used to create calibration curves. Quality assurance and control studies were conducted to ensure the accuracy and reliability of the device. In this context, UME CRM 1201 (spring water) was analysed as certified reference material (CRM). When the results obtained in CRM measurements were compared with certified values, it was determined that the accuracy rates ranged from 90% to 100% on an element basis, and the analytical precision was within $\pm 10\%$ error limits. Additionally, a 1 ppm internal standard (Agilent 5188-6525) was continuously fed into the system throughout the measurements, and the performance stability of the instrument was monitored. The standardisation studies conducted supported the reliability of the element concentrations obtained from the river samples.

Preparing Samples for Analysis

Sediment samples taken from stations identified along the Karasu River in Sinop Province were subjected to preliminary preparation procedures in the laboratory. The samples were dried at 105 °C, then passed through a 0.77 μm (200 mesh) sieve and homogenised. Approximately 0.22–0.26 g of each sample was weighed, and solutions were prepared to bring the final volume to 50 mL. As a result of these procedures, dilution ratios ranging from 190 to 231 were obtained. Thus, the Karasu River sediment samples were prepared in accordance with standard pre-treatment procedures and made ready for the ICP-MS analysis.

Sediment Pollution Indices

If iron (Fe) had been found among the metals analysed, Fe would have been used as the reference metal in the index calculations. However, since Fe analysis was not performed in the current study, aluminium (Al), which was among the elements analysed, was taken as the reference value. Although Al does not fall into the heavy metal category, it was included in the study to enable calculations to be made and indices to be interpreted consistently.

Enrichment Factor (EF): A normalised indicator used to determine whether a metal is anthropogenic (human-induced) or derived from natural processes [25].

$$EF = \frac{\frac{E_0}{Fe_0}}{\frac{E_s}{Fe_s}} \quad (1)$$

E_0 : Measured value of the element,

E_s : Standard value of the element,

$Fe_{\text{ö}}$: Measured value of Fe,

Fe_s : Standard value of Fe [26, 27].

Geo-accumulation Index (I_{geo}): Developed by Müller, this index classifies metal accumulation on a logarithmic scale to define the degree of contamination in the environment [25].

$$I_{geo} = \frac{En}{1.5 Bn} \quad (2)$$

En : Element measured value,

Bn : Element reference value.

Contamination Degree (C_d): The sum of the C_f values of all metals, indicating the overall contamination level of the station under investigation [28].

$$C_d = \sum_{i=1}^n C_f \quad (3)$$

The contamination factor for each element (i) under investigation is denoted by C_{Fi}.

Contamination Factor (C_f): The ratio of the measured concentration of a metal to its background value, which determines the degree of contamination caused by a single metal [25].

$$CF = \frac{C_{\text{sample}}}{C_{\text{background}}} \quad (4)$$

Very low pollution (CF < 1), moderate pollution (1 . CF . 3), considerable pollution (3 . CF . 6) and very high pollution (CF > 6) [29].

Modified Contamination Factor (mCdeg): Modified Contamination Factor is a multielement index used to assess soil and sediment contamination. Calculated by dividing the C_d value by the number of metals examined, it facilitates the comparison between different regions or stations.

$$mC_d = \frac{1}{n} \sum_{i=1}^n CF_i \quad (5)$$

where C_f is the contamination factor for each element (i) separately, and n is the number of elements examined [30, 31].

Table 4. Scales and threshold values [32]

Contamination Factor (Cf)						
$Cf < 1$	$1 \leq Cf < 3$	$3 \leq Cf < 6$	$Cf \geq 6$			
Low	Moderate	Considerable	Very high			
Contamination Degree (Cd)						
$Cd \leq 8$	$8 \leq Cd \leq 16$	$16 \leq Cd \leq 32$	$Cd \geq 32$			
Low	Moderate	Considerable	Very high			
Modified Contamination Degree (mCd)						
$mCd < 1.5$	$1.5 \leq mCd < 2$	$2 \leq mCd < 4$	$4 \leq mCd < 8$	$8 \leq mCd < 16$	$16 \leq mCd < 32$	$mCd \geq 32$
Nil to very low	Low	Moderate	High	Very high	Extremely high	Ultra high
Enrichment Factor (EF)						
$EF < 2$	$2 \leq EF < 5$	$5 \leq EF < 20$	$20 \leq EF < 40$	$EF \geq 40$		
Minimal	Moderate	Significant	Very high	Extremely high		
Geoaccumulation Index (Igeo)						
$Igeo \leq 0$	$0 < Igeo < 1$	$1 \leq Igeo < 2$	$2 \leq Igeo < 3$	$3 \leq Igeo < 4$	$4 \leq Igeo < 5$	$Igeo \geq 5$
Practically uncontaminated	Uncontaminated to moderately	Moderately	Moderately to strongly	Strongly	Strong to extremely	Extremely

Findings

Sediment Analysis Results

The data obtained as a result of the analyses of sediment samples collected from Karasu Stream between April and September 2023 are presented in the graphs and tables below. In this study, ten different heavy metals (chromium (Cr), cobalt (Co), nickel (Ni), copper (Cu), zinc (Zn), cadmium (Cd), aluminum (Al), arsenic (As), lead (Pb) and uranium (U)) were analyzed in sediment samples. These data are expressed as monthly measured values with standard errors at three stations (Station 1, Station 2 and Station 3).

Table 5. Monthly analysis results of heavy metals examined in sediment samples taken from Karasu Stream (g/kg).

		Al	Cr	Co	Ni	Cu	Zn	As	Cd	Pb	U
April	1. St.	6053.494	17.124	2.826	14.258	3.828	15.417	1.759	0.042	2.462	0.175
April	2. St.	16103.871	41.660	6.863	31.877	9.494	25.299	2.828	0.074	5.788	0.309
April	3. St.	12143.526	28.001	4.111	22.052	6.975	22.982	1.815	0.054	3.863	0.214
May	1. St.	11977.220	26.966	4.355	25.557	7.289	15.436	1.585	0.054	4.206	0.243
May	2. St.	19101.491	40.010	6.575	32.210	10.802	24.387	4.059	0.089	7.366	0.363
May	3. St.	13665.881	31.971	5.864	26.268	7.698	18.728	2.957	0.072	5.257	0.348
June	1. St.	9515.336	26.721	4.428	22.084	7.625	15.983	2.392	0.070	4.827	0.323
June	2. St.	22599.510	52.316	7.714	40.886	12.762	29.620	3.755	0.096	7.137	0.425
June	3. St.	19331.989	41.297	6.731	34.645	11.417	22.565	2.917	0.078	6.742	0.364
July	1. St.	4600.049	17.162	2.432	9.986	3.522	10.926	1.810	0.037	2.330	0.233
July	2. St.	24181.863	51.136	8.346	38.972	14.339	27.943	4.159	0.092	12.164	0.470
July	3. St.	19204.405	39.843	5.941	32.797	11.249	23.323	2.099	0.080	8.979	0.301
August	1. St.	14231.445	34.994	6.529	28.961	8.202	19.478	4.790	0.065	5.269	0.313
August	2. St.	27941.430	53.685	9.804	44.209	16.329	30.010	2.563	0.109	10.018	0.495
August	3. St.	2502.816	51.200	9.093	37.149	14.379	27.806	4.471	0.094	8.958	0.428
September	1. St.	10853.523	30.636	4.252	21.280	6.453	14.252	1.735	0.054	4.234	0.264
September	2. St.	15224.148	42.275	6.515	29.229	10.617	21.317	3.916	0.073	5.866	0.461
September	3. St.	11303.857	31.207	5.595	25.405	8.381	17.974	2.050	0.066	5.284	0.382

As seen in Table 5, the highest concentrations of all heavy metals we examined were measured at Station 2 (Sinop Airport location), which is located near the settlement. The lowest heavy metal concentrations were measured at Station 1 (Çobanlar Village), which is generally close to the stream source. When the heavy metal concentrations in the Karasu Stream sediments were examined, it was observed that the most abundant heavy metal in all stations was aluminum (Al) and the least abundant one was cadmium (Cd).

Macro Elements

Statistical analysis of the heavy metal aluminum (Al), one of the macro elements examined in the sediment samples in this study, was obtained as a result of samples collected from three stations between April and September 2023. The average of the results obtained is shown in Table 6 and Figure 2.

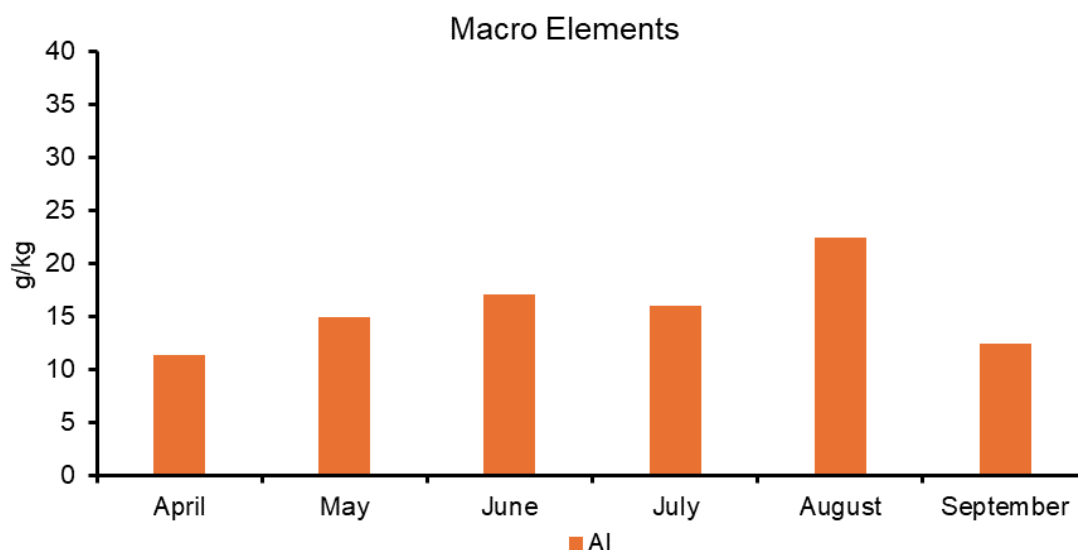


Figure 2. Monthly analysis results of macro element (Al) in sediment samples collected from Karasu Stream

The highest aluminum concentration was measured in August. It is thought to occur due to the decrease in precipitation, temperature, agricultural activities and population increase in August.

Trace Elements

Statistical analysis of the heavy metals chromium (Cr), cobalt (Co), nickel (Ni), copper (Cu), zinc (Zn), arsenic (As) and lead (Pb) from the trace elements examined in the sediment samples in this study was obtained as a result of samples collected from the three stations between April and September 2023. The mean values of the obtained results are presented in Table 7 and illustrated in Figure 3.

Table 7. Monthly analysis results of trace elements in sediment samples collected from Karasu Stream

Trace Elements (mg/kg)						
Elements	April	May	June	July	August	September
Cr	28.93±3.55 ^c	32.98±1.90 ^{bc}	40.11±3.71 ^{ab}	36.05±5.02 ^{bc}	46.63±2.93 ^a	34.71±1.90 ^{bc}
Ni	22.73±2.55 ^c	28.01±1.06 ^{bc}	32.54±2.77 ^{ab}	27.25±4.41 ^{bc}	36.77±2.20 ^a	25.30±1.15 ^{bc}
Zn	21.23±1.50 ^{ab}	19.52±1.31 ^{bc}	22.72±1.97 ^{ab}	20.73±2.55 ^b	25.76±1.61 ^a	17.85±1.03 ^c
Cu	6.77±0.82 ^c	8.60±0.56 ^{bc}	10.60±0.77 ^{ab}	9.70±1.61 ^b	12.97±1.23 ^a	8.48±0.60 ^{bc}
Pb	4.04±0.48 ^c	5.61±0.47 ^{bc}	6.24±0.36 ^{ab}	7.82±1.45 ^a	8.08±0.72 ^a	5.13±0.24 ^{bc}
Co	4.60±0.60 ^c	5.60±0.33 ^{bc}	6.29±0.49 ^b	5.57±0.86 ^{bc}	8.48±0.50 ^a	5.45±0.33 ^{bc}
As	2.13±0.17 ^c	2.87±0.36 ^{bc}	3.02±0.20 ^b	2.69±0.37 ^{bc}	3.94±0.35 ^a	2.57±0.34 ^{bc}

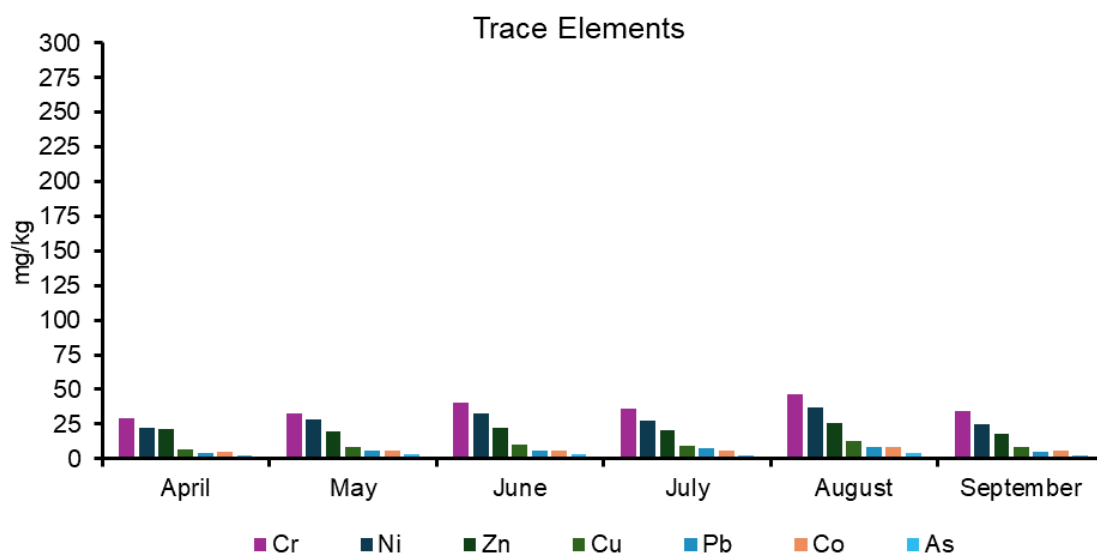


Figure 3. Monthly analysis results of trace elements in sediment samples collected from the Karasu Stream

The highest concentrations of chromium (Cr), cobalt (Co), nickel (Ni), copper (Cu), zinc (Zn), arsenic (As) and lead (Pb) were measured in August. This is thought to be associated with the decrease in precipitation, temperature, agricultural activities and population density in August.

Vestige Elements

Statistical analysis of the trace elements in the sediment samples in this study, cadmium (Cd) and uranium (U), was obtained from samples taken at three stations between April and September 2023. The average of the results obtained is shown in Table 6 and Figure 4.

Table 6. Monthly analysis results of trace elements in sediment samples collected from the Karasu Stream

Vestige Elements ($\mu\text{g/kg}$)						
Element	April	May	June	July	August	September
U	232.49 $\pm 19.93^c$	317.89 $\pm 18.92^b$	370.61 $\pm 15.01^{ab}$	334.83 $\pm 35.24^b$	411.74 $\pm 26.55^a$	368.86 $\pm 28.64^{ab}$
Cd	56.64 $\pm 4.70^c$	71.67 $\pm 5.03^{bc}$	81.26 $\pm 3.91^{ab}$	69.29 $\pm 8.32^{bc}$	89.68 $\pm 6.45^a$	64.16 $\pm 2.75^c$

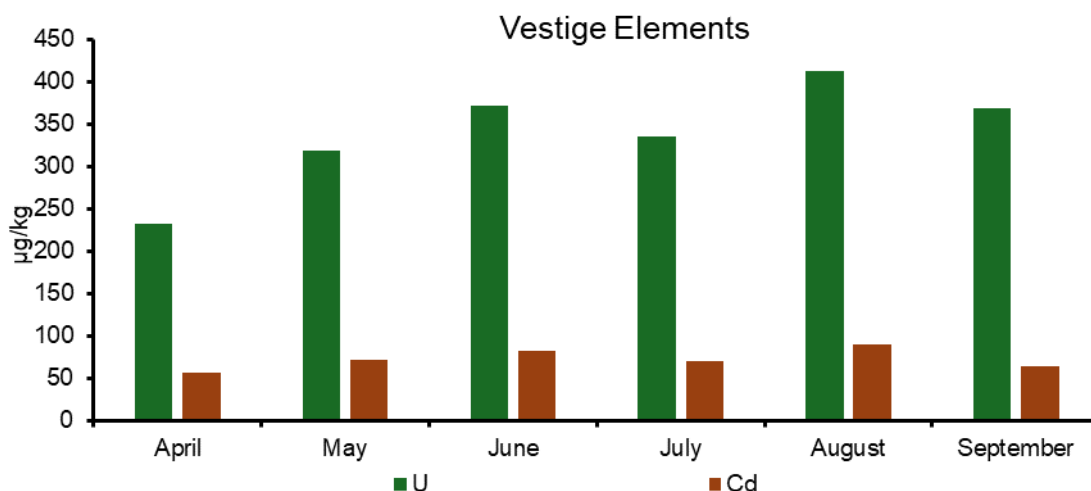


Figure 4. Monthly analysis results of trace elements in sediment samples collected from the Karasu Stream

The highest concentrations of cadmium (Cd) and uranium (U) were measured in August. This phenomenon is thought to be linked to lower precipitation and temperature levels, along with decreased agricultural activities and population density in August.

Pollution Indices

According to the analysis findings, the pollution factor (Cf) values for Al, As, Cd, Co, Cr, Cu, Pb, Zn and U elements generally remained below 1, and these metals were classified in the low pollution category. However, the Ni element reached a value of 2.15, particularly at the second station, indicating a moderate pollution level. This finding reveals that, despite the overall low pollution level, Ni shows a notable degree of accumulation at certain stations. The pollution level (Cd) values calculated on a station basis were found to be 3.36 at Station 1, 6.03 at Station 2, and 4.87 at Station 3. The fact that Cd values remain below 8 at all stations indicates that the region's overall pollution level is low and that heavy metal accumulation does not pose an ecological risk. The modified pollution degree (mCd) values at the stations were determined to be 0.33, 0.60 and 0.49, respectively. These values remained well below the 1.5 limit at all stations and showed that heavy metal pollution was negligible. Additionally, the mCd results show that there are no significant differences between stations and that the region exhibits a generally homogeneous pollution level. The enrichment factor (EF) values of most metals in the study area were found to be below 2, and these metals were placed in the minimal enrichment category. Only the Ni element showed moderate enrichment, with values in the 2–3 range. This result indicates that Ni exhibits a relatively higher accumulation tendency compared to natural background values, while other metals do not show significant enrichment. The geo-accumulation index (Igeo) values were negative for most metals, and these metals were classified as uncontaminated. Only Ni was classified as slightly contaminated, with values ranging from 0.2 to 0.5 at some stations. The negative Igeo values obtained for Cr and Cu indicate completely clean conditions.

Statistical Calculations and Results

According to the results of the hierarchical cluster analysis, the monthly distributions of sediment heavy metal concentrations in the Karasu Creek were grouped into three main clusters. April (1) and September (6) showed close similarity and were included in the same cluster. May (2), June (3), and July (4) formed a second cluster, demonstrating that heavy metal accumulation during the summer period were similar. In contrast, August (5) was clearly distinct from all other periods and was considered an independent cluster (Figure 5). This finding indicates the existence of seasonal variation in Karasu Creek. The weathering observed particularly in August may be due to hydrometeorological factors such as high temperatures, decreased flow rates, and increased evaporation, as well as agricultural activities and anthropogenic inputs. Overall, the hierarchical cluster analysis indicates that heavy metal distributions in sediments are not homogeneous throughout the year, with distinct clusters occurring during the spring, summer, and autumn transitions.

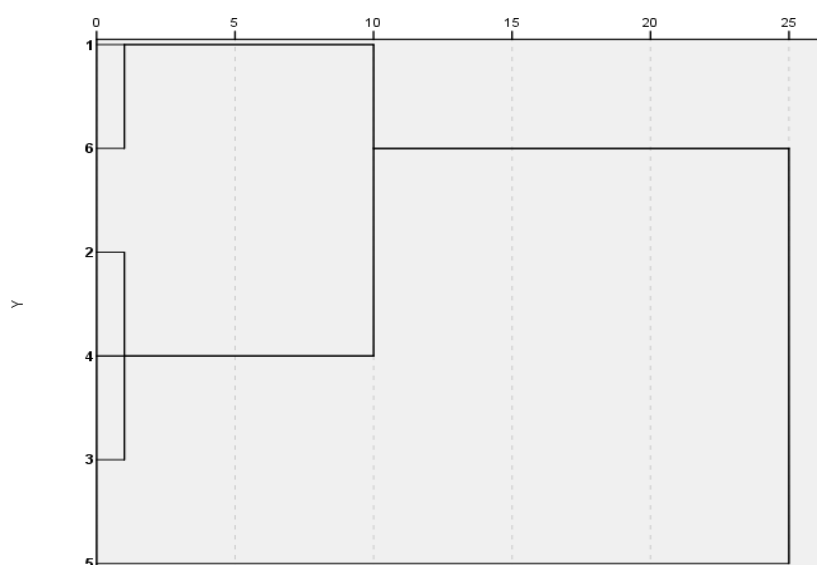


Figure 5. Monthly hierarchical cluster analysis results

Pearson correlation analysis, performed to determine the relationships between heavy metals in the Karasu Creek sediment samples, revealed highly positive and statistically significant relationships among many elements. Particularly notable were the strong correlations between Al and Cu ($r=0.976$; $p=0.001$), Al and Cd ($r=0.977$; $p=0.001$), and Al and As ($r=0.968$; $p=0.002$). Similarly, highly significant relationships were observed between Cu and Cd ($r=0.947$; $p=0.004$) and Ni and Cu ($r=0.967$; $p=0.002$). This suggests that these elements exhibit similar geochemical behavior and are likely transported into the sediment environment from the same sources (e.g., geogenic processes or anthropogenic inputs). In contrast, correlations between Zn and other metals were weaker and mostly not statistically significant (e.g., Zn-Ni, $r=0.800$; $p=0.056$). This suggests that Zn may have different accumulation mechanisms than other metals. Furthermore, correlations between U and other elements are relatively low, showing

significant associations only with Cr ($r=0.876$; $p=0.022$) and As ($r=0.816$; $p=0.048$). This result suggests that uranium may be particularly associated with regional geological structures or specific hydrogeochemical processes (Figure 6). In general, high correlation coefficients indicate that heavy metals are sourced from common sources, while low correlations indicate the influence of different processes. These findings provide important evidence that heavy metal accumulation in sediments is shaped both by natural (geological structure) and anthropogenic factors (agriculture, industry, domestic waste).

		Correlations									
		Al	Cr	Co	Ni	Cu	Zn	As	Cd	Pb	U
Al	Pearson Correlation Sig. (2-tailed) N	1 6									
Cr	Pearson Correlation Sig. (2-tailed) N	,948 ,004 6	1 6								
Co	Pearson Correlation Sig. (2-tailed) N	,961 ,002 6	,963 ,002 6	1 6							
Ni	Pearson Correlation Sig. (2-tailed) N	,967 ,002 6	,959 ,003 6	,953 ,003 6	1 6						
Cu	Pearson Correlation Sig. (2-tailed) N	,976 ,001 6	,991 ,000 6	,961 ,002 6	,967 ,002 6	1 6					
Zn	Pearson Correlation Sig. (2-tailed) N	,836 ,038 6	,757 ,081 6	,794 ,059 6	,800 ,056 6	,766 ,076 6	1 6				
As	Pearson Correlation Sig. (2-tailed) N	,968 ,002 6	,945 ,004 6	,986 ,000 6	,966 ,002 6	,957 ,003 6	,746 ,089 6	1 6			
Cd	Pearson Correlation Sig. (2-tailed) N	,977 ,001 6	,923 ,009 6	,926 ,008 6	,984 ,000 6	,947 ,004 6	,868 ,025 6	,940 ,005 6	1 6		
Pb	Pearson Correlation Sig. (2-tailed) N	,857 ,029 6	,806 ,053 6	,750 ,086 6	,750 ,086 6	,865 ,026 6	,579 ,228 6	,767 ,075 6	,772 ,072 6	1 6	
U	Pearson Correlation Sig. (2-tailed) N	,740 ,093 6	,876 ,022 6	,804 ,054 6	,799 ,056 6	,851 ,032 6	,354 ,491 6	,816 ,048 6	,693 ,127 6	,684 ,134 6	1 6

Figure 6. Monthly correlation analysis results

According to the results of the single-sample t-test, the mean values of all heavy metals measured in the Karasu Creek sediments were found to be statistically significant ($p<0.05$). The significance level was calculated as 0.000 for all elements Al, Cr, Co, Ni, Cu, Zn, As, Cd, Pb, and U, and these values revealed that the metal accumulations in the sediment were not random but rather were clearly present. Particularly high levels of Al (Avg.=15725.6 mg/kg), Zn (Avg.=21.30 mg/kg), and Ni (Avg.=28.76 mg/kg) indicated that these metals were concentrated in the sediments. Furthermore, the $p<0.05$ significance level obtained for relatively low levels of elements such as Cd (Avg.=0.07 mg/kg) and U (Avg.=0.33 mg/kg) also demonstrated systematic accumulation of these elements. These results demonstrate that heavy metal accumulation in the Sinop Karasu Stream poses a statistically significant environmental problem, with certain metals (Ni, Zn, Al) in particular showing significant accumulation. When the findings are evaluated together with both clustering and correlation analyses, it can be concluded that metal pollution in sediments is influenced by both seasonal dynamics and potential anthropogenic inputs.

Discussion and Conclusion

This study, which examined heavy metal pollution in the sediments of the Karasu Stream, covers the period from April to September 2023. Potential pollution sources that may affect the water quality of the Karasu Stream include sewage water originating from domestic wastewater (heavy metals originating from detergents, cleaning products and personal care products), discharge of water leaking from septic tanks into the creek without treatment, transportation of fertilizers and pesticides used in agricultural activities into the creek through drainage, discharge of wastewater from facilities located on the creek side (e.g. workshops and businesses); and pollutants originating from exhaust gases, heavy metals and dust carried by atmospheric deposition (industrial emissions, traffic-related pollutants, long-distance transport, forest fires). When the sediment analysis results were examined, the highest concentrations of all heavy metals were measured at Station 2 (Sinop Airport location), which is situated near residential areas. This shows that the station, which is located near the residential area, is strongly influenced by human activities. At Station 3 (Dibekli village Akliman location), which is close to the area where the Karasu Stream meets the sea, the second highest pollutant concentrations were measured. Station 3 lies within the Akliman settlement area in the Sinop city center and is under the influence of the region. Although it is not as close to the settlement as Station 2, it is not as far from human activities as Station 1. It is thought that this situation caused the heavy metals examined to give average results. Pollutant concentrations are lower at Station 1, which is closer to the stream source. This is due to the fact that Station 1 (Çobanlar Village) is relatively far from human activities and where natural conditions are more dominant. Significant variations in sediment quality were observed across the different stations. Especially in terms of heavy metals, Station 2 differed significantly from other stations. Heavy metals from these pollution sources can accumulate in sediments, posing threats to both aquatic ecosystems and human health. In particular, considering the population density of the settlements through which Karasu Stream passes and the types of fertilizers and pesticides (organophosphates, organochlorides, pyrethroids, neonicotinoids) used in agricultural areas, it is thought that the sediment quality of Karasu Stream may have been negatively affected. Özbay et al. [33] investigated the concentration of heavy metals aluminum (Al), lead (Pb), zinc (Zn), manganese (Mn), cadmium (Cd), iron (Fe), chromium (Cr), nickel (Ni) and copper (Cu) in the sediment samples taken seasonally from the Berdan Stream within the borders of Mersin. When the results were examined, the heavy metal concentrations were listed as $Fe > Al > Mn > Ni > Cr > Zn > Cu > Pb > Cd$. It was concluded that the Berdan Stream was under threat of pollution. Kır et al. [34] analyzed some heavy metals in sediment and water in Kovada Lake between 05.04.2005 and 23.02.2006. It was observed that the highest heavy metal density in water was iron (Fe). Heavy metal concentrations were also found to reach their highest levels during spring and winter. It was observed that the highest heavy metal density in sediments was aluminum (Al). Heavy metal and microplastic pollution is a serious environmental problem on both a local and global scale, according to research conducted in different parts of the world. According to

Pandit et al. [14] 10 different metal concentrations have increased significantly in Bangladesh over the past 20 years, with As, Pb, Cd, Cr, Fe and Mn values in particular exceeding threshold values. According to research conducted by Wang and Chadwick [15], Cd, Cr, and Hg levels in the Huaihe and Haihe rivers in China were 20-40 times higher than the permitted limits. Chen et al. [19] noted that As, Cr, and Pb posed serious health problems for people living along the Yangtze River, while Liang et al. [18] emphasised that Cd, As, and Pb were widespread in sediments in the Dianchi Basin. For the illustration of regional differences in metals, Han et al. [20] identified high concentrations of Cd and Pb in the Dagu River and presented a concentration ranking of $As > Cd > Cu > Pb > Zn > Cr$. Baycan et al. [21] This demonstrated that new pollutants other than heavy metals also posed significant threats to aquatic ecosystems. According to Basti et al. [16], organic carbon played a role in the mobility of heavy metals and showed that pollution was affected by both internal sediment properties and external inputs. These global findings were supported by studies conducted in Turkey. Kutlu et al. [22] detected Cr, Ni, and Pb accumulations in the sediments of the Pülümür River and emphasised that these metals caused moderate to high levels of ecological concern. Özşeker and Terzi [23] demonstrated that rainfall-runoff conditions influenced the distribution of metals and that Cd and Pb reach significant seasonal levels in the Değirmendere River Basin. From the review of all these studies, it is clear that heavy metals and microplastics pose major threats to ecosystem health and human health, but the most prominent metals and pollution levels vary from place to place. Additionally, two important factors affecting pollution levels are source-based pollution (agriculture, mining, industry, wastewater) and sediment dynamics (organic carbon, seasonality, particle size). Therefore, it is clear that sustainable management solutions must address sediment processes and new-generation pollutants holistically, in addition to reducing pollution. When we review literature, heavy metal pollution was examined in water and sediment samples found in aquatic environments such as rivers, lakes, ponds, streams and dams, which is similar to the study we conducted. As a result, in parallel to the findings we obtained, aluminum (Al) concentrations were generally measured as high and cadmium (Cd) concentrations were measured as low. The analyses revealed that the Cf values of Al, As, Cd, Co, Cr, Cu, Pb, Zn, and U were below 1 and were classified as very low pollution, whereas only Ni was classified as medium pollution with a value of 2.15. This indicates that Ni is a prominent pollutant on a regional scale. Indeed, Kutlu et al. [22] reported similar findings in the Pülümür River, while Özşeker and Terzi [23] reported higher Ni values compared to other metals in the Değirmendere River. In our study, Cd values ranged from 3.36 to 6.03 at the stations and remained below the threshold of 8, indicating very low overall pollution. Similarly, Liang et al. [18] and Wang and Chadwick [15] reported that Cd values in different river basins mostly remained in the low risk class. The calculated mCd values (0.33–0.60) remained well below 1.5 at all stations, indicating that heavy metal contamination was at a negligible level. This result is consistent with the definition by Abraham and Parker [31] that areas with $mCd < 1$ are considered clean or lightly impacted regions. EF values remained below 2 in most metals, indicating minimal

enrichment, with only Ni showing moderate enrichment in the 2–3 range. Loska et al. [26] have indicated that EF values > 5 in areas exposed to industrial effects are a serious indicator of anthropogenic pressure; accordingly, the low EF values in our study indicate that the pressure in the region is limited. Igeo results were also negative for most metals, which were classified as ‘uncontaminated,’ with only Ni in the 0.2–0.5 range being classified as ‘slightly contaminated.’ This classification coincides with the thresholds proposed by Balık and Tunca [32] and is also consistent with the findings of Islam et al. [17], which showed that Ni has higher Igeo values than other metals in the transboundary river basin. All these data suggest that the region generally carries a low risk of heavy metal pollution, but Ni stands out as a common problem both in literature and in the results of this study. This study provided important information about the sediment quality of Karasu Stream and showed that the stream ecosystem was affected by human activities. The findings emphasize that necessary measures should be taken for the protection and improvement of Karasu Stream.

The following measures can be taken to reduce heavy metal pollution in Karasu Stream:

Regular Inspection of Industrial Wastes: One of the most important reasons for heavy metals mixing into streams is industrial waste. These wastes must be regularly inspected and cleaned with appropriate treatment systems.

Limiting Chemical Use in Agriculture: Incorrect use of fertilizers and pesticides has the potential to introduce heavy metals into water supplies. Therefore, environmentally friendly farming methods should be encouraged and chemical use should be limited.

Waste Management and Recycling: Proper management of solid waste and industrial waste prevents heavy metals from mixing into water. Furthermore, widespread recycling practices also help reduce pollutants.

Treatment of Industrial and Domestic Wastes: Domestic and industrial wastes must be treated before being discharged into streams. Water treatment plants must be operated effectively and wastes must be prevented from being discharged directly into water resources.

Education and Awareness Raising: Educating local people and industrial enterprises on environmental protection is an important step in combating heavy metal pollution. Everyone needs to act consciously to prevent pollution.

Ecosystem Monitoring and Control: It is important to regularly monitor streams, identify pollutant sources and carry out effective controls to eliminate these sources. Water quality should be continuously monitored with environmental monitoring programs.

Legal Regulations and Penal Sanctions: Effective legal regulations should be established to address heavy metal pollution, and punitive measures should be enforced against those who fail to comply. These measures can help protect ecosystem health by reducing heavy metal pollution in streams.

When the results obtained are evaluated as a whole, it was determined that heavy metal pollution in the study area was at a very low level, with only Ni showing a slight tendency towards contamination and

enrichment at some stations. All other metals remained at low contamination and minimal enrichment levels, indicating that the region poses a low ecological risk. The TEL value (≈ 20.9 mg/kg) outlined in the Sediment Quality Guides (SQG) is often exceeded by the Ni concentrations found in Kara Su Creek, which vary from 22.73 to 36.77 mg/kg. The August measurement of 36.77 mg/kg came close to but fell short of the PEL (≈ 51.6 mg/kg) limit [35]. This implies that, although the risk level is still below the critical threshold, Ni deposition in Karasu Creek should be taken into account from an ecotoxicological standpoint. In a similar vein, Li et al. [36] found that Ni levels rose, particularly in regions with high agricultural activity, and that Ni accumulation was substantially connected with pH and total organic carbon (TOC). Furthermore, Chung et al. [37] investigated the distribution of heavy metals, such as Ni, and the ecotoxic consequences of sediment elutriates in the Andong Basin, highlighting the potential ecological risks at concentrations that are close to the PEL values. These results imply that it would be suitable to track Ni levels in the Karasu Stream in accordance with seasonal variations and bolster them with long-term data. In this study, the monitoring results conducted in Karasu Creek, when evaluated together with previous studies conducted in streams, lakes, and coastal areas in the Sinop region, indicate that heavy metal levels in the area are generally low to moderate. In a study conducted on Akgöl Reservoir by Emin and Mutlu [38], the water quality was reported to be Class I according to the Surface Water Quality Regulation in terms of analyzed heavy metals and did not pose a risk to the ecosystem. Furthermore, Bat et. al. [39] conducted a study on the Sinop coast and examined the accumulation of various metals, particularly Zn and Cu, in sediment, water, and polychaete samples. They revealed that seasonal differences in the sediments were not evident. These findings suggest that the results obtained in Karasu Creek in this study are consistent with regional trends and that pollution has not yet reached critical levels. Hierarchical cluster analysis of Karasu Stream sediments revealed significant seasonal variations in heavy metal accumulations by month. The inclusion of April and September in the same cluster suggests that similar hydrological conditions in spring and early autumn shaped the metal distributions. The fact that May, June, and July formed separate clusters suggests that metal concentrations exhibited a common trend during the summer period. Conversely, the inclusion of August in a single, independent cluster suggests that heavy metal accumulations during this period differed from those of other months and may have been influenced by factors such as high temperature, low flow, or increased agricultural activity. Pearson correlation analysis findings also support these interpretations. The analysis revealed highly positive correlations between elements such as Al, Cu, Cd, and As, suggesting that these metals are fed from common sources or exhibit similar geochemical behavior in the sediment environment. In contrast, the weaker correlations of elements such as Zn and U with other metals suggest that these elements may originate from different sources or have accumulation processes related to unique environmental conditions. Therefore, when both clustering and correlation analyses are evaluated together, it can be concluded that heavy metal accumulations in

Karasu Stream sediments are shaped by both seasonal hydrological dynamics and different source inputs.

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Author Contributions The authors contributed equally to this work.

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