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EVALUATION OF HEAVY METAL ACCUMULATION IN SEDIMENT, SURFACE WATER AND SOME PLANTS IN THE KARASU RIPARIAN ZONE

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ABSTRACT. This study aims to determine and compare the heavy metal concentrations in the water, sediment, and plants of the Karasu river in Erzurum, Türkiye. In this context, aluminum (Al), manganese (Mn), iron (Fe), nickel (Ni), zinc (Zn), and barium (Ba) concentrations were measured for this study. *Salix excelsa* (willow), *Rosa canina* L., *Pyrus elaeagnifolia* (wild pear), *Malus sylvestris* (wild apple), and *Hippophae rhamnoides* L. (sea buckthorn) plants were used as samples. Heavy metal concentrations follow the order of $Al^{3+} > Fe^{2+} > Mn^{2+} > Ba^{2+} > Ni^{2+} > Zn^{2+}$ in the sediment. Heavy metal concentrations follow the order of $Al^{3+} > Fe^{2+} > Ba^{2+} > Mn^{2+} > Zn^{2+} > Ni^{2+}$ in water. The leaf part of *Salix excelsa* has the highest bioconcentration factor (BCF) for Mn among plant parts. Mean BCF values were as follows: $Zn^{2+} > Mn^{2+} > Ba^{2+} > Ni^{2+} > Al^{3+} \approx Fe^{2+}$. In addition, the highest plant translocation factor (PTF) values were determined in *Salix excelsa* for Al and Fe; in *Rosa canina* L. for Ni and Zn; in *Hippophae rhamnoides* L. for Mn and in *Malus sylvestris* for Ba.

KEY WORDS: Heavy metals accumulation, Sediment, Water, Plant, Riparian Zone

INTRODUCTION

Heavy metal contamination has received global attention due to its irreversible impact on the ecosystem and its ability to propagate through the food chain [1, 2]. Due to their pollution and buildup, toxicity, non-biodegradable qualities, the abundance, and cumulative nature, they generate significant issues [3, 4]. Natural resources, such as rock erosion, factories, and human-induced activities, such as fertilizer and pesticide usage in agriculture, can contribute to heavy metal pollution [5]. In inhabited regions, rivers are natural conduits for the movement of polluted sediment. Terrestrial metals from geological or anthropogenic sources collect in sediments by flowing rivers or streams [6]. Toxic chemicals, such as heavy metals and persistent organic pollutants, frequently bond to fine-grained solid carriers, particularly clay minerals and organic compounds. Additionally, they can be deposited in sediments and riverside vegetation along waterways [7]. Due to the discharge of untreated residential and industrial wastes, urban rivers produce a rise in heavy metal concentrations in river water and are linked with water quality issues [8, 9]. During transit through the river environment, heavy metals may undergo several transformations due to occurrences such as dissolution and precipitation, among others. In addition to water, information regarding the total metal concentrations of sediment, which is an integral and dynamic component of the river basin, must be obtained [10-12]. Nevertheless, sediment analysis alone is insufficient for determining the environmental impact of its contamination. In fact, the heavy metals absorbed by plants through their roots accumulate in the food chain and constitute a significant hazard to the health of animals and humans. Industrial pollutants, such as those emitted by the cement plants, which are also regarded as the primary source of pollutants, inflict significant harm to ecosystems. Cement manufacturers are regarded as significant emitters of dust and heavy metals such as Cd, Cr, Hg, Pb, and Zn [13-15]. Some metals, such as Cu and Zn, are vital to life because they are required in several metabolic systems, but Cd, Pb, and Cr may be hazardous in large amounts. When they contact the biological system, heavy metals such as lead, mercury, arsenic, copper, zinc, and cadmium are very poisonous [16].

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Heavy metal uptake by plants is a complex process governed by several interdependent elements, including plant species, genotype, soil metal mobility, and soil characteristics [17]. The accumulation of heavy metals in plants varies with plant species and the efficiency of metal absorption, which is determined by soil-to-plant transfer factors [18]. Hence, it is essential to assess the quantities and distribution of heavy metals in other river ecosystem components (flora, fauna, etc.).

Biomonitoring is often utilized to give a quantitative evaluation of the environmental quality and to determine the degree of exposure to contaminants in an ecosystem [19].

This study aims to assess the heavy metal (Al^{3+}, Mn^{2+}, Fe^{2+}, Ni^{2+}, Zn^{2+}, and Ba^{2+}) status of selected plant species cultivated in the water, sediment, and riparian vegetation of the Karasu River and to identify plants that can be utilized as biomonitors in riparian zone restoration. The distribution of heavy metals in environments makes it advantageous to employ certain species in large-scale monitoring studies. Particularly, the usage of tolerant plants in restoration research will boost the efficacy of healing and rehabilitation efforts.

EXPERIMENTAL

Description of study area

The Karasu Sub-basin is located in the north of the Euphrates-Tigris basin. The basin is named after the Karasu River, which is one of the main tributaries of the Euphrates River. Aziziye, Çat and Aşkale districts of Erzurum Province are within the boundaries of Karasu Lower Basin. The drainage area of the Karasu River, the most significant tributary of the Euphrates River, in Erzurum is 1642 km^2 and its average flow rate is 4,304 m^3/sec [20, 21]. The research area is approximately 40 km away from the city center of Erzurum. In the research area, 3 sampling locations were chosen at 1 km intervals. The altitude of the research area is 1700 meters, and the region has a continental climate characterized by cold, snowy winters and hot, dry summers. Sampling points are located between 4420998 North 653931 East coordinates (Figure 1). Agriculture, livestock, domestic wastes, slaughterhouse wastes, and sugar plant waste products are believed to be the primary contributors to river pollution.

Chemicals and reagents

The chemicals used in the study were all analytical or ACS grade and mostly used for the preparation of samples or as consumables during instrumental analysis. TOC/TN calibration solutions were prepared by KHP (99.5%) (Merck KGaA., Darmstadt, Germany), Na_2CO_3 (99.9%) (Merck KGaA., Darmstadt, Germany) and HCO_3 (99.7%) (Sigma-Aldrich Corp., St. Louis, USA), KNO_3 (99%) (Merck KGaA., Darmstadt, Germany). Anion calibration solution (F^-, Cl^-, NO_2^-, NO_3^-, SO_4^{2-}) was Dionex Seven Anion Standard (Thermo Fisher Scientific Inc., California, USA) which was used as purchased. Homogenization solution chemicals were 5-sulfosalicylic acid (99%), EDTA (98.5%), $C_6H_7NaO_6$ (98%) (all from Sigma-Aldrich Corp., St. Louis, USA). Sample digestion chemicals for elemental analysis were HNO_3 (65%), H_2O_2 (30%) and HCI (37%) (all from Merck KGaA., Darmstadt, Germany).

Instruments and apparatus

The pH and EC of the water samples were determined by the test device (Model: WTW MultiLine P4, Germany). Color was measured by Spectroflex UV/VIS 6600 (WTW, Germany). Turbidity was measured by Micro 100 turbidimeter (HF Scientific Inc., USA). TOC (TC-IC), TC and TN analyzes were performed with Shimatzu TOC (Model: TOC-L CPN) and TN (Model: TNM-L ROHS) (all from Shimatzu Corporation, Kyoto, Japan) devices. High purity (99.9999%) dry air was used as a carrier gas. Anion analysis was performed by ion-chromatograph system Dionex, ICS-3000 (Dionex Corporation, USA). The elemental analyses of sediment samples were carried out with the Agilent 7800 ICP-MS (Agilent Technologies, Inc., USA) device, using the procedures for plant samples. Also, Field Emission Scanning Electron Microscope (SEM, Zeiss Sigma 300) (Zeiss company, Germany) coupled with an energy-dispersive X-ray fluorescence spectrometer (EDX: INCA Energy Instruments, England) was used to examine the sediment surface and elemental mapping images. In addition, sediment samples were mineralogically characterized using a Panalytical Empyrean brand X-ray diffraction (XRD) (Malvern Panalytical Ltd., UK) device between 10-90^0. The results of the analysis were reported via the X 'Pert High Score program. Milestone Ethos UP brand microwave system (Milestone Srl., İtaly) was used for the combustion procedure.

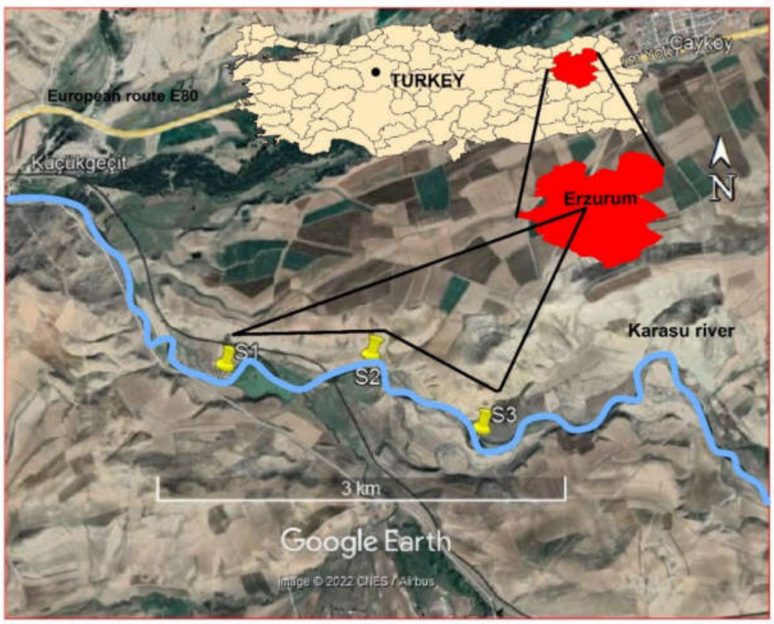


Figure 1. Location of the study area.

Water and sediment sampling and preparation

A total of 12 samples (9 water and 3 sediments) were collected in May 2022 (spring season). Samples were collected from three different stations (S1-S3). The water samples were collected via 500 mL polyethylene terephthalate (PET) bottles from the riverside, stored at 4 °C, and transported to the laboratory for analysis.

Surface sediment samples (0–5 cm depth) were gathered using a stainless steel sampler. By mixing sediments that were randomly collected from the sample locations, sediment samples were created. Until the analysis, samples were put in glass containers and kept in a cooler at 4 °C. The sediment samples were dried at 105 °C to a constant weight and grounded in the mortar grinder after being stored at room temperature for 24 hours before analysis. Sediment samples were passed through a sieve of < 63 μm and placed in plastic bags for chemical analysis [11, 22, 23]. The pH and EC of the sediment samples were determined in a 1:2.5 soil/water solution.

Plant sampling and preparation

Five plant samples, namely *Salix excelsa* (willow), *Rosa canina* L., *Pyrus elaeagnifolia* (wild pear), *Malus sylvestris* (wild apple), and *Hippophae rhamnoides* L. (sea buckthorn) were picked with a stainless steel blade. Sediment sample locations were also used to collect samples from plants. Zipped bags were used to collect plant specimens from branches and leaves. At each location, a uniform sample of three plants of the same species was gathered. The plant leaves were washed with distilled water to eliminate particles and then dried at 70 °C until constant weight. The dried plant samples were grounded and sieved through a 100 mm porous sieve. Plant samples were then homogenized in a 5 vol/g solution containing 2% 5-sulfosalicylic acid, 1 mM EDTA, and 0.15% sodium ascorbate. Before the combustion process, the required sample amount (about 0.3 g) was measured on a precision scale. In the Milestone Ethos Up microwave system, the combustion procedure required for analysis were carried out. All samples were kept in the microwave for 1 hour by adding 3 ml of nitric acid, 1 ml of hydrogen peroxide, and 6 ml of hydrochloric acid to the leaf samples and 9.9 ml of nitric acid and 0.1 ml of hydrochloric acid to the branch samples. Following the combustion phase, the samples were diluted with distilled water (1:100) and analyzed by ICP-MS [23].

Bioconcentration factor (BCF) and translocation factor (PTF) were calculated for each plant based on plant and soil metal concentrations to determine if plants could be classified as accumulators. BCF is defined as the ratio of heavy metal concentration in plants to soil [16, 23-26]. Plant bioconcentration factor (BCF) and plant translocation factor (PTF) were calculated using the following equation:

$$\mathrm{BCF}_{\mathrm{concentration}} = \mathrm{C}_{planttissue}/\mathrm{C}_{sediment}$$

$$\mathrm{PTF}_{\mathrm{translocation}} = \mathrm{BCF}_{plantbranch}/\mathrm{BCP}_{plantleaves}$$

where $C_{concentration}$ = metal concentration in plant tissue, $C_{sediment}$ = metal concentration in sediment, $C_{plantleaves}$ = metal concentration in plant leaves, $C_{plantbranch}$ = metal concentration in plant branch. Plants with bioconcentration and translocation factors higher than 1 can be used as bio accumulators. In addition, plants can be used as phytostabilizers if BCF is >1 and PTF < 1, and as phytoextractors if BCF is < 1 and PTF > 1 [27, 28]. The data were analyzed using IBM SPSS Statistics 20.0 software.

RESULTS AND DISCUSSION

Characteristics of the water and sediment samples

Some characteristics of the water and sediment which was used as a sample are given in Table 1.

Table 1. Some characteristics of sediment and water samples.

Parameter	Water	Sediment
EC (µS/cm)	366	749
pH	8.11	8.47
Turbidity (NTU)	28	-
Color (Pt-Co)	31	-
Fluoride (mg/L)	1.15	Non-detected
Chloride (mg/L)	248.3	22.3
Nitrate-N (mg/L)	7.99	1.1
Sulphate (mg/L)	29.13	63.9
TOC (mg/L)	41.13	605.4
TC (mg/L)	44.03	620.1
TN (mg/L)	1.88	749.2

It was concluded that there were kaolinite, quartz (SiO_2), dolomite, and calcium carbonate in the sediment samples (Figure 2).

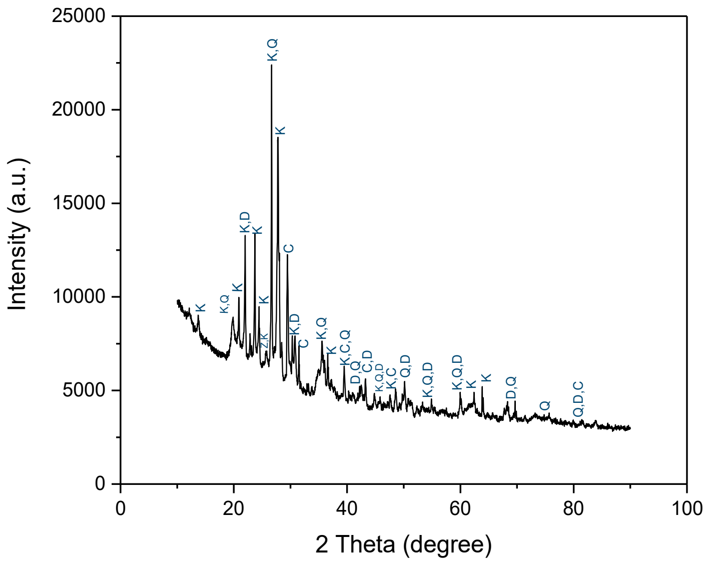


Figure 2. XRD pattern of sediment samples (K; Kaolinite (98-006-8697), Q; Quartz (98-003-1228), D; Dolomite (98-017-1524), C; Calcium Carbonate (98-042-3568)).

Heavy metal contents in water samples

It is crucial to determine whether toxic heavy metals such as Al, Mn, Fe, Ni, Zn, and Ba reach humans through the food chain. The concentration ranges of the heavy metals found in the sample locations were as follow: Al (31.81-71.99 ppb), Mn (10.98-26.94) ppb, Fe (63.18-37.15 ppb), Ni (5.39-1.84 ppb), Zn (3.70-9.51 ppb) and Ba (28.24-23.67 ppb), respectively. The highest mean value was observed in Al (56.33 ppb), and the lowest mean value was observed in Ni (3.52 ppb). The mean values of heavy metal contents in water samples followed the order of $Al^{3+} > Fe^{2+} > Ba^{2+} > Mn^{2+} > Zn^{2+} > Ni^{2+}$. Whereas, the maximum concentration of Ni (5.39) and Mn (26.94) was found in the SW1 sample. (Table 2). The mean values of heavy metals in the water samples were below WHO, World Bank (WB), and US EPA standards [29]. The highest concentration was observed in Al. Aluminum concentration varied depending on water pH. In fact, aluminum concentrations in neutral and flowing waters were often below 1.0 mg/L [30].

The recommended acceptable limits for Ni, which is harmful to human health, in wastewater and agricultural soils are 0.02 and 0.05 ppm, respectively. The mean concentration value of Ni in the water samples was <4 ppb and this was lower than the standards set by WHO, World Bank (WB), and US EPA [29]. The mean concentration of nickel in the water sample collected from SW3 alone was 5.39 ppb. The existing urban wastewater treatment plant's favorable impact on water quality can be used to explain why the study's results were significantly below the specified limit values. Besides, another study [31] also supports this conclusion. The mean concentration of Ba at the sample locations did not exceed the limit values. Even though there are studies with high levels of Ba in well water, there is insufficient evidence of the carcinogenic toxicity of barium intake through drinking water [32-34]. For this reason, it is believed that periodic and seasonal monitoring of the region will be helpful.

Table 2. Heavy metal concentration in water

Sample	Al^{3+}	Mn^{2+}	Fe^{2+}	Ni^{2+}	Zn^{2+}	Ba^{2+}
SW1	51.68	10.98	44.75	1.84	6.15	23.67
	61.81	14.75	51.19	2.63	4.26	24.55
	62.27	26.94	37.15	2.48	5.62	27.57
SW2	42.79	25.09	60.90	4.10	5.70	26.80
	70.98	24.26	52.11	3.75	5.43	28.24
	51.37	23.83	52.43	4.13	9.51	26.59
SW3	62.26	25.09	51.57	5.39	6.44	27.36
	31.81	23.78	48.15	3.03	3.70	27.03
	71.99	23.36	63.18	4.27	4.37	26.80
Mean ± SD	56.3±13.19	22.01±5.37	51.27±7.81	3.52±1.11	5.69±1.70	26.51±1.47
Min.	31.81	10.98	37.15	1.84	3.70	23.67
Max.	71.99	26.94	63.18	5.39	9.51	28.24

Heavy metal contents in sediment samples

SEM images and EDX analyses of the sediment sample were shown in Figure 3. Moreover, Al, Mn, Fe, Ni, and Ba elements in sediment were observed in EDX analysis. For the sediment sample, the means of element percentages were in the descending order of Si > C > Al > Fe > Ba ≈ Ni ≈ Mn.

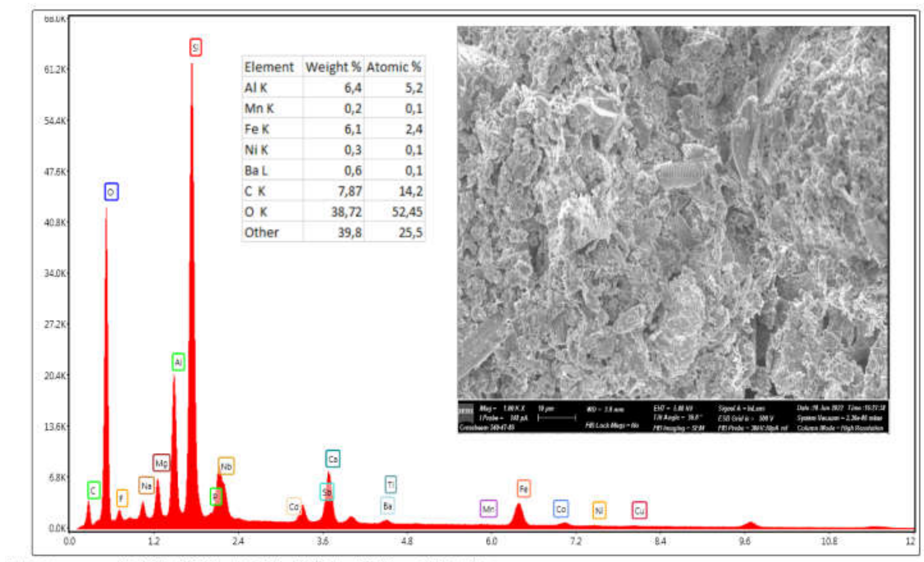


Figure 3. SEM images and EDX analysis of the sediment sample.

Heavy metal contents in the sediment were presented in Table 3. The mean concentrations of heavy metals in the sediment were Al (70.28 ppm), Fe (70.18 ppm), Mn (2.54 ppm), Ba (0.79 ppm), Ni (0.27 ppm) and Zn (0.26 ppm), respectively.

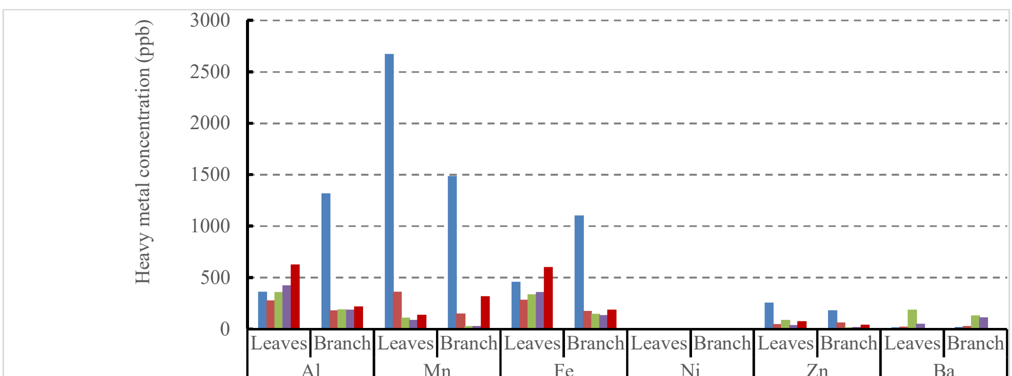
Table 3. Heavy metal concentration in sediment.

Sample	Al^{3+}	Mn^{2+}	Fe^{2+}	Ni^{2+}	Zn^{2+}	Ba^{2+}
SS1	65.54	1.31	51.22	0.11	0.11	0.75
SS2	72.15	3.15	74.15	0.45	0.46	0.98
SS3	73.16	3.17	65.16	0.25	0.20	0.65
Mean ± SD	70.28±4.14	2.54±1.07	65.16±6.35	0.27±0.17	0.26±0.18	0.79 ± 0.17
Min.	65.54	1.31	51.22	0.11	0.11	0.65
Max.	73.16	3.17	94.15	0.45	0.46	0.98

The maximum values of Al (73.16 ppm) and Mn (3.17) were observed at the SS3 sample point. It is also thought that the high amount of Al may be due to the structure of clay (Kaolinite-$H_4Al_2O_9Si_2$) (Figure 2). Ba and Fe were detected at the SS2 sampling point. The trend of heavy metal contents of soil was $Al^{3+} > Fe^{2+} > Mn^{2+} > Ba^{2+} > Ni^{2+} > Zn^{2+}$. In this context, the concentration values and EDX analysis results were likewise consistent.

Heavy metal contents in plant

The concentrations of Al, Fe, Mn, Ba, Ni, and Zn vary based on plant species and plant parts.



	Al Leaves	Al Branch	Mn Leaves	Mn Branch	Fe Leaves	Fe Branch	Ni Leaves	Ni Branch	Zn Leaves	Zn Branch	Ba Leaves	Ba Branch
Salix excelsa	361.8	1319.8	2671.5	1487.9	458.4	1104.0	9.3	5.0	258.6	181.4	17.0	19.2
Rosa canina L.	278.0	182.8	364.1	152.3	283.0	174.9	6.7	6.3	50.7	64.8	23.9	28.8
Pyrus elaeagnifolia	358.8	191.1	110.1	30.6	340.3	148.9	4.8	3.4	86.8	17.4	187.1	131.4
Malus sylvestris	423.6	186.1	89.0	30.4	358.9	133.7	5.6	4.8	39.2	20.5	52.8	115.2
Hippophae rhamnoides	624.9	219.4	138.5	319.0	601.4	188.0	8.6	2.1	77.0	41.2	8.7	7.1

Figure 4. Variation of heavy metal concentrations of plants by species and plant parts.

Willow leaves contained the highest Mn content (2672 ppb), while its branches contained the highest Al and Mn concentrations. It is known that the majority of heavy metals taken up by Willow species from their roots are transferred to their leaves [35]. Willows are widely utilized for phytoremediation of soil contaminated with heavy metals because they exhibit high resistance to these contaminants [36]. The branch part of *Seabuckthorn* contained the lowest amount of Ni (2.1 ppb) (Figure 4). At the same time, the leaves of other plants held high levels of Mn, but the Sea buckthorn only possessed high levels of Mn in its branches. Except for Mn, all heavy metal concentrations were found to be elevated in the leaves of sea buckthorn. In a study on sea buckthorn, the mineralization of heavy metals in the leaves was described as an excessive and gradual accumulation [37, 38]. The highest Ni content was detected in the leaf of all plant species. Only the branch portion of Willow was discovered to have the greatest content of Al and Fe when plant parts were analyzed. Zn, on the other hand, contained a higher concentration of heavy metals in the leaf part than in the branch part of plants except for *Rosa canina* L. The maximum concentration of Ba was observed in wild pear. In the wild apple, Ba was only discovered in high concentrations in the branch. Upon comparing sediment, plant, and water samples, it was shown that the concentrations of Mn and Zn in the willow's leaves and branches were higher than those in sediment and water. According to a study conducted in the same location, sediment samples contained higher levels of heavy metals than water [21, 38]. In general, willow contained the highest concentrations of all heavy metals except for Ba (which was only discovered in wild pear).

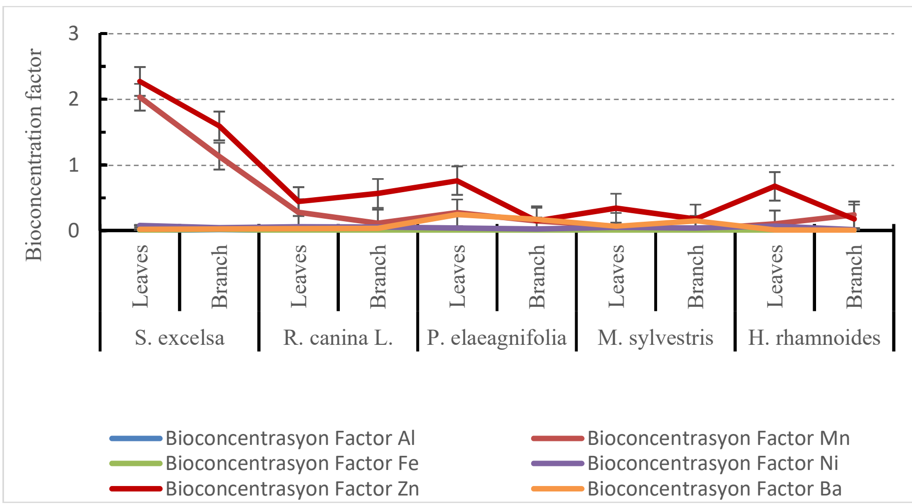


Figure 5. BCFs of Al, Mn, Fe, Ni, Zn, and Ba in plant species. Error bars indicate standard deviation.

As was the case with concentrations, bioconcentration factors differed by plant species and organs. BCF value was determined as >1 only for Mn and Zn. Mean BCF values of *S. excelsa* were found to be 2.04 in the leaf and 1.13 in branches for Mn and 1.02 in the leaf for Zn (Figure 5). Considering the data, *S. excelsa* might be classified as a bio accumulator plant (BCF > 1).

The translocation factors of heavy metals were presented in Figure 6. The majority of translocation factors greater than 1 were reported for Al (3.65) and Mn (2.30). The highest translocation factors for all metals were found for Al (*S. excelsa*), Fe (*S. excelsa*), Mn (*H. rhamnoides*), Ba (*M. sylvestris*), Zn (*R. canina* L.), Ba (*R. canina* L.) and Ba (*S. excelsa)*, respectively. These findings indicate that Al heavy metal and *S. excelsa* species are primarily capable of transporting heavy metals from branches to leaves.

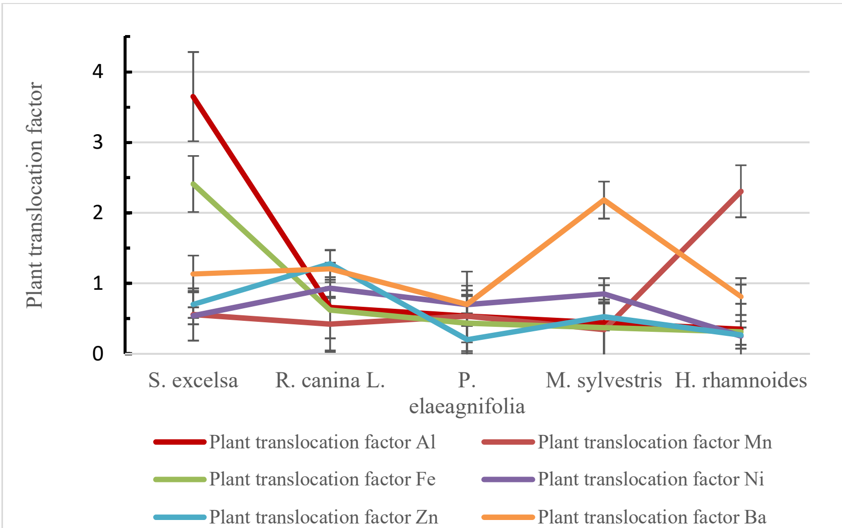


Figure 6. Translocation factors of heavy metals in plant tissues (branch-leaves). Error bars indicate standard deviation.

CONCLUSION

In this study, the content and distribution of metals in water, sediment, and several plants were determined. Al has the highest concentration in water, whereas Ni has the lowest concentration. However, the concentrations did not exceed the permissible level. The favorable effect of the biological wastewater treatment plant is believed to have contributed to this outcome. However, periodic and seasonal monitoring is required in particular. The metal concentrations in the sediment samples were within the acceptable range. Al and Fe concentrations were found to be greater than those of other elements in sediment samples. The amounts of heavy metals in plants varied both among themselves and between plant parts. *Salix excelsa* and *Hippophae rhamnoides* plants had higher concentrations of heavy metals. The bioconcentration factor (BCF) was highest in the leaf part of *Salix excelsa* species for Mn. It has been demonstrated that plant translocation factors (PTF) for heavy metals and plant species vary between plant species. PTF was highest for Al in the leaf part of *Salix excelsa* species. Even if the environmental status of the study area is generally favorable, it must be monitored to avoid it from becoming risky in the future, and the riparian zones must be planted with appropriate species.

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