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# GEOCHEMICAL DISTRIBUTION OF ELEMENTS IN WATERS AND SEDIMENTS OF THE PLESENSKA RIVER WITH A SPECIAL FOCUS ON THE PRESENCE OF HEAVY METALS

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## Abstract

This paper presents the data from laboratory analyses of water and sediments in autumn and early spring (September 2024 and February 2025). The analyses included waters and sediments from the Plesenska River and the confluence of the Kiselica River with the Zletovska River, as well as a spring located in the immediate vicinity of the Plesenska River. Water analyses were conducted to assess the actual condition and geochemical distribution of elements during the two studied periods, with a special focus on the presence of heavy metals. To obtain additional information about the components transported by the water flow and to gain insight into the cause-and-effect relationships and mechanisms, sediment examinations were also performed.

**Key words:** *water, sediments, distribution, pH, heavy metals.*

## INTRODUCTION

Geographically, the research area covers the northeastern part of the Republic of North Macedonia, specifically extending from the village of Plesinci to the northwest and continuing southeast of the Municipality of Probistip, at a distance of approximately 4-10 km (Figure 1). The research area belongs to the Kratovo-Zletovo volcanic region, which is part of two neotectonic morphostructures, the Osogovo Horst and the Probistip-Kocani Graben.

The separate lithological formations in this area belong to two regional geotectonic units, the Serbian-Macedonian massif and the Vardar zone, which show wide occupancy of the Balkan Peninsula. The boundary between these geotectonic units in the area of the Osogovo Horst is represented by the Ratkovica-Plackovica thrust, along which the mass of the Serbian-Macedonian massif is thrusted over Paleozoic schists from the Vardar zone [1, 2].

## MATERIAL AND METHODS

### Regional and geotectonic position of the research area

The studied locality is part of the Kratovo-Zletovo volcanic area, which, with its complex structural-geological and metallogenetic composition, is an integral part of the Lece-Chalkidiki metallogenetic zone (Figure 2).

The Lece-Halkidiki zone starts from the mining region of Lece in Serbia to the northwest and extends southeast to eastern Halkidiki in Greece [3]. The average width of the zone is 30-35 km, with a length of several hundred kilometers. Spatially, the metallogenetic zone of Lece-Halkidiki is localized in the boundary area between the Vardar zone and the Serbian-Macedonian massif [3].

Figure 1. Geographic location of the research area

Mineralization in this metallogenetic zone is distributed across several smaller metallogenetic units: Lece, Lojane-Nikustak Kratovo-Zletovo, Damjan-Bucim-Borov Dol, which are spatially located, both in the Serbian-Macedonian massif and the Vardar zone.

The Kratovo-Zletovo volcanic area is characterized mostly with disjunctive as well as plicative tectonics. It has been further fractured by faults, with the direction of these faults being NW-SE and ENE-WJW. These fault structures acted as conduits for the circulation of ore-bearing solutions. They are associated with Pb/Zn, polymetallic, and U-mineralization, whose ore deposits are localized in the volcanic calderas formed during the Tertiary intermediate calc-alkaline magmatism.

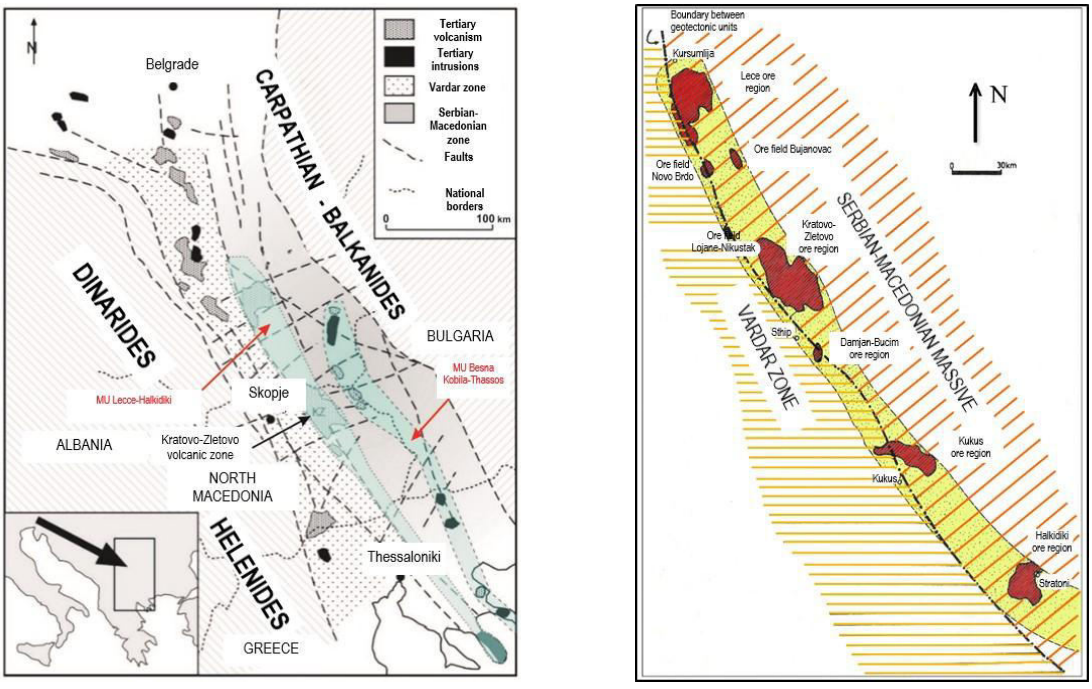

Figure 2. Regional, geotectonic, and metallogenetic position of the Lece-Halkidiki zone [3]

**Geological characteristics of the research area and its wider vicinity**

According to the "Basic Geological Map of the SFRY 1:100 000" sheets Kratovo (K 34-69) [4], and Stip (K 34-81) [5], and the explanations for them, the geological structure of the research area and its broader surrounding is represented by Tertiary rocks and Quaternary sediments (Figure 3).

Their age ranges from Upper Eocene, Miocene, Pliocene, Pliocene-Quaternary, Pleistocene, and Holocene.

The **Upper Eocene** is represented by the upper zone of flysch: clays and sandstones. The most dominant members are sandy clays composed of illitic clay material and detritus in the form of quartz, feldspar, muscovite, heavy minerals such as garnets, tourmalines, metallic minerals, sphene, rutile, and others. According to the mineral composition, the sandstones correspond to subgraywackes and graywackes.

The **Miocene** is represented by dacitic ignimbrites, as well as laporites, tuffaceous sandstones, and clays. The dacitic ignimbrites contain fragments of dacites and pyroclastic material, followed by gneisses, schists, gabbros, and others. The laporites, tuffaceous sandstones, and clays lie above the dacitic ignimbrites, while volcanic tuffs and breccias have been deposited on top of them. Due to the fact that in some places they are pierced by andesites, Miocene sediments are hydrothermally altered and opalized.

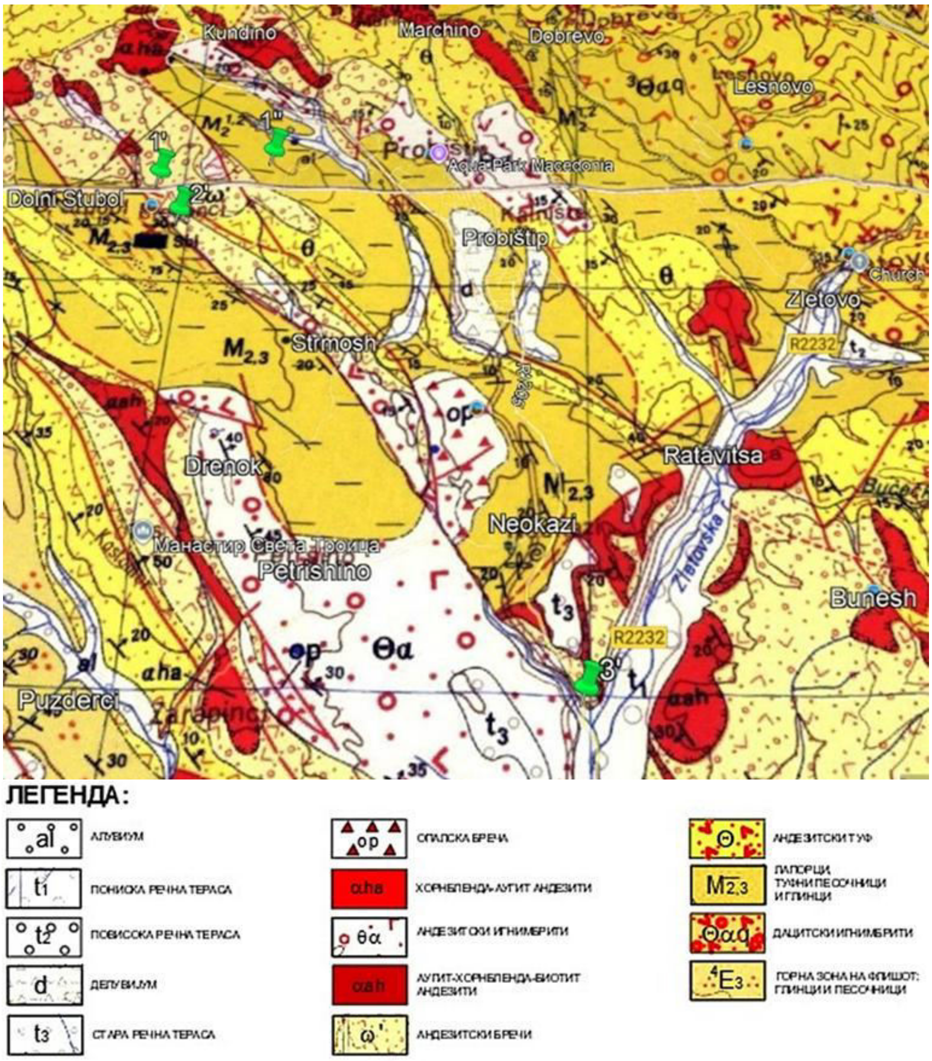


Figure 3. Geological structure of the research area and its wider vicinity with locations of sampling (Basic Geological Map of Macedonia 1:100 000, sheets Stip K 34-81, 1965/68 and Kratovo K 34-69, 1963/68) [4, 5]

**The Pliocene** is represented by andesitic tuff, which is widespread over the Miocene and Upper Eocene sediments. The size of the fragments that make up the tuffs varies, ranging from a few millimeters to several tens of centimeters. The tuffs are intensely altered and fragmented. The main mass is composed of volcanic ash and fragmented hypidiomorphic grains of plagioclase, biotite, and, less commonly, pyroxenes, amphiboles, and magnetite, as well as considerable chloritic and limonitic material.

**The Pliocene-Quaternary** is represented by andesitic breccias and augite-hornblende-biotite andesites. The andesitic breccias lie directly above the tuffs and are composed of unprocessed fragments of andesites, chaotically scattered in the main mass, which is made up of volcanic ash and broken mineral grains. Augite-hornblende-biotite andesites occur exclusively in the andesitic breccias. In their main mass, phenocrysts of plagioclases, oligoclase-andesine, as well as hypidiomorphic and idiomorphic grains of pyroxene-augite, opaque grains of hornblende with prismatic elongated shapes, and less frequently biotite are present, and they are affected by alterations.

**The Pleistocene** is represented by andesitic ignimbrites, hornblende-augite andesites, and opal breccia. The andesitic ignimbrites occur directly above the volcanic breccias or the uppermost limestones, where they are often fragmented and very crumbly. They are composed of phenocrystals and a matrix that acts as cement.

Hornblende-augite andesites lie above the andesitic breccias and ignimbrites. These are altered and weathered, composed of phenocrystals of plagioclases, hypidiomorphic grains of pyroxene, hornblende, and biotite, which is quite fresh, along with a matrix.

The opal breccia is characterized by an intense red color and a crystal-clastic structure. In places, silex has formed, and pronounced silicification is observed.

**The Holocene** is represented by an old river terrace, deluvium, higher river terrace, lower river terrace, and alluvium.

The old river terrace is the oldest and is mainly composed of andesite gravels, less commonly quartz and gneisses.

Deluvium represents a fragmented-loose cover composed of Paleogene sediments. The higher river terrace developed as an erosion-accumulation terrace along the valley of the Zletovska River, at a height of 20 to 50 meters above the riverbed, and is almost exclusively made up of loosely bound gravels and sandstones.

The lower river terrace is present along the valley of the Zletovska River at a height of 5 to 10 meters above the riverbed. It is mostly composed of clays, silts, sandy silts, and gravels.

Alluvium is present in the valleys of certain river courses and is mainly composed of gravels and sands [1].

**Research Methodology**

At the very beginning, a sampling and research plan was developed, and then actions were taken based on it. Within the research, field, laboratory, and cabinet activities were carried out.

**The field activities** consisted of collecting water and sediment samples from the Plesenska River and the Kiselica River, as well as spring water from a faucet located in close proximity to the Plesenska River. The samples were collected in two different time periods, specifically in the autumn (September 2024) and in the early spring (February 2025).

The samples of running water were collected from locations where the river had a slower flow. Water was filled into clean plastic bottles of 0.5-1 l, labeled with the appropriate marking and sample number (Figure 4).

The sediment samples were collected from the riverbeds of the aforementioned rivers and packed in clean plastic bags (Figure 4), labeled with the appropriate marking and sample number. The exact location of each sampling site was recorded with GPS unit GARMIN etrex 10.

The locations of the collected samples with precise coordinates are shown in Figure 5 and in Table 1.

Figure 4. Sampling of water and sediments


Figure 5. Locations of water and sediment sampling [6]

Table 1. Coordinates of the water and sediment sampling locations

| Locations and coordinates of water and sediment testing | | | | |
|---|---|---|---|---|
| Location | September 2024 | February 2025 | Coordinates | |
| | | | Y | X |
| | Sample mark | | | |
| Plesenska River - Faucet | 1" | 1" | 7 595 800 | 4 651 800 |
| Plesenska River – above the village | 1' | 1' | 7 594 369 | 4 651 495 |
| Plesenska River – below the village | 2' | 2' | 7 594 690 | 4 650 928 |
| Confluence of the Kiselica River into the Zletovska River | 3' | 3' | 7 599 761 | 4 644 955 |

The laboratory analyses of the water and sediment samples were performed by the ICP-MS (Inductively Coupled Plasma - Mass Spectrometry).

The desk activities involved a detailed analysis of the obtained results and their computer processing, including the creation of correlation diagrams and their interpretation.

Relevant foreign and domestic available literature and documentation were also used [7-10].

## RESULTS AND DISCUSSION

The comparison of element concentrations in the water samples was performed according to reference values in the "Decree on Water Classification (Official Gazette of the Republic of Macedonia No. 18/99)" for classes I and II [11].

Since there is currently no specific legal regulation in the Republic of North Macedonia for treating soil from multiple environmental aspects, and no legally established limit values for harmful and dangerous substances in soils, the comparison of element concentrations in sediment samples was conducted according to the national substrate concentration for the Netherlands and Environmental Chemistry of the Elements [12], as referenced from (JICA, 2008) [10].

The results of the laboratory tests of the water and sediment samples are presented in Table 2 and Table 3, respectively.

Table 2. Concentration of elements in water samples (September and February 2024/25) according to the MAC for water classes I and II, expressed in μg/l

| Element | Unit | Water sample mark | | | | | | | | MAC I and II class [μg/l] |
|---|---|---|---|---|---|---|---|---|---|---|
| | | (1") | | (1') | | (2') | | (3') | | |
| | | Sept. | Febr. | Sept. | Febr. | Sept. | Febr. | Sept. | Febr. | |
| As | μg/l | 3,9 | 3,5 | 8,7 | 76,9 | 2,7 | 1,2 | 2,5 | <1 | 30 |
| Cu | | 2,6 | 17,7 | 4,6 | 1,3 | 7,7 | 35,9 | 4,5 | 1,0 | 10 |
| Bi | | 7,7 | <1 | 1,2 | <1 | <1 | <1 | 1,1 | <1 | 50 |
| B | | 36,7 | <10 | 39,3 | <10 | 37,5 | 13,7 | 58,9 | 25,4 | 200 |
| Fe | | 574 | 7152 | 3186 | 14023 | 3191 | 5032 | 654 | 18,7 | 300 |
| Cd | | <1 | 4,3 | 1,2 | <1 | 1,1 | 2,9 | <1 | <1 | 0,1 |
| K | | 4154 | 2545 | 3922 | 3824 | 12483 | 5014 | 8892 | 3843 | nd |
| Ca | | 29266 | 62481 | 46405 | 44538 | 45710 | 53528 | 113460 | 81032 | nd |
| Co | | 81,6 | 147 | 48,5 | 175 | 58,8 | 134 | 39,0 | <1 | 100 |
| Mg | | 5165 | 3880 | 7153 | 6946 | 10078 | 6474 | 22080 | 15687 | nd |
| Mn | | 30,7 | 1181 | 286 | 567 | 489 | 1613 | 674 | 1411 | 50 |
| Na | | 9696 | 15738 | 19981 | 11846 | 22821 | 25415 | 26443 | 120341 | nd |
| Ni | | 3,3 | 28,6 | 11,2 | 35,9 | 13,6 | 27,4 | 10,0 | 1,9 | 50 |
| Pb | | 2,3 | 3,5 | 1,8 | 3,9 | 2,3 | 2,4 | 5,8 | <1 | 10 |
| Ag | | 2,3 | <1 | 1,3 | <1 | 1,4 | <1 | 4,4 | <1 | 2 |
| Tl | | <1 | 6,8 | 1,1 | 5,8 | 1,0 | 3,6 | 1,5 | <1 | 3 |
| Ti | | <1 | 1,6 | 1,1 | 2,9 | 1,3 | 1,6 | 1,0 | 1,5 | 100 |
| Cr | | 4,3 | 2,0 | 6,0 | 3,0 | 3,1 | 2,1 | 1,2 | <1 | 50 |
| Zn | | 38,6 | 448 | 157 | 651 | 194 | 412 | 166 | 340 | 100 |
| pH | | 2,8 | 2,9 | 2,7 | 3,4 | 3,5 | 3,1 | 5,8 | 5,4 | 6,3-8,5 |

Note - nd – no MAC (maximum allowed concentration) value

Table 3. Concentration of elements in sediment samples (September and February 2024/25) according to the national substrate concentration for the Netherlands and Environmental Chemistry of the Elements [12], expressed in mg/kg

| Element | Unit | Sediment sample mark | | | | | | | | Substrate - Netherland [mg/kg] | Average soil Bowen [mg/kg] |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | (1") | | (1') | | (2') | | (3') | | | |
| | | Sept. | Febr. | Sept. | Febr. | Sept. | Febr. | Sept. | Febr. | | |
| Al | | 12801 | 59119 | 13692 | 73183 | 15794 | 42095 | 25204 | 22718 | - | - |
| Sb | | 2,1 | <1 | <1 | 1,2 | 1,7 | <1 | 6,6 | 4,9 | - | - |
| As | | 19,4 | 68,2 | <1 | 122 | 17,2 | 23,2 | 12,8 | 31,1 | 29 | 6 |
| Cu | | 40,9 | 23,5 | 43,2 | 31,4 | 43,9 | 14,1 | 70,2 | 32,9 | 36 | 30 |
| Ba | | 1099 | 609 | 2,8 | 808 | 1119 | 575 | 1871 | 471 | - | - |
| Be | | 2,8 | 0,7 | 3,2 | 0,8 | 2,8 | 0,5 | 3,7 | 0,4 | - | - |
| Bi | | 3,3 | <1 | 1,3 | 1,1 | 1,6 | <1 | 1,3 | 2,1 | - | - |
| B | | 4,9 | 27,4 | 13,6 | 106 | 4,2 | 5,4 | 2,6 | 9,4 | - | - |
| V | | 110 | 118 | <1 | 146 | 90,6 | 75,1 | 98,3 | 40,6 | - | - |
| S | | 13108 | 4590 | 7329 | 7049 | 14746 | 3007 | 10493 | 1584 | - | - |
| Ga | | <1 | 7,7 | <1 | 9,5 | <1 | 5,9 | <1 | 4,0 | - | - |
| Ge | | <1 | <1 | <1 | <1 | <1 | <1 | <1 | <1 | - | - |
| Fe | | 17437 | 42289 | 15558 | 50495 | 12730 | 21618 | 10666 | 17261 | - | - |
| Cd | | 1,4 | <1 | <1 | <1 | 1,5 | <1 | 2,5 | 2,9 | 0,80 | 0,35 |
| K | | 20730 | 12872 | 20235 | 14294 | 16670 | 8743 | 11108 | 5323 | - | - |
| Ca | | 3261 | 3467 | 5369 | 3014 | 4025 | 2979 | 59831 | 3177 | - | - |
| Sn | mg/kg | 2,9 | 1,6 | 1,0 | 1,8 | 2,5 | 1,6 | 4,8 | 2,0 | - | - |
| Co | | 3,3 | 3,6 | 3,8 | 4,3 | 4,5 | 2,1 | 16,4 | 10,4 | 9 | 8 |
| Li | | 15,2 | 6,5 | 13,1 | 8,4 | 11,2 | 5,0 | 16,8 | 5,0 | - | - |
| Mg | | 3150 | 2579 | 2914 | 3388 | 2598 | 1302 | 11229 | 1496 | - | - |
| Mn | | 113 | 118 | 3,1 | 128 | 77,3 | 34,8 | 1125 | 18315 | - | 1000 |
| Mo | | 3,5 | 1,5 | <1 | 1,7 | 3,1 | 1,5 | 4,1 | 2,5 | - | - |
| Na | | 4623 | 4780 | 6470 | 5292 | 5150 | 3839 | 8772 | 2379 | - | - |
| Ni | | 2,2 | 3,0 | 3,6 | 2,3 | 3,7 | <1 | 54,8 | 8,5 | 35 | 50 |
| P | | 2914 | 613 | 22,2 | 833 | 3319 | 577 | 5926 | 525 | - | - |
| Pb | | 52,4 | 46,3 | 4,3 | 65,6 | 54,2 | 48,6 | 171 | 568,2 | 85 | 35 |
| Se | | 3,6 | 3,7 | <1 | 4,1 | 2,6 | 2,5 | 3,2 | 1,9 | - | - |
| Ag | | 2,6 | <1 | 2,3 | <1 | 2,5 | <1 | 0,9 | 1,4 | - | - |
| Sr | | 1454 | 498 | 28,6 | 582 | 1217 | 403 | 1550 | 134 | - | - |
| Tl | | 10,7 | 2,8 | <1 | 3,5 | 10,9 | 1,5 | 5,7 | 1,2 | - | - |
| Ti | | 5247 | 718 | 7288 | 873 | 5266 | 147 | 7100 | 841 | - | - |
| Cr | | 33,6 | 6,6 | 26,2 | 12,8 | 20,3 | 5,4 | 63,3 | 6,7 | 100 | 70 |
| Zn | | 52,7 | 20,7 | 24,4 | 25,4 | 39,6 | 28,9 | 394 | 173 | 140 | 90 |

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From Table 2, it can be concluded that all water samples are characterized by a low, or acidic pH value, which allows greater ability to dissolve and mobilize elements, specifically heavy metals.

Based on the results of the water sample analyses, correlation plots (Figures 6, 7 and 8) were constructed for both studied time periods (September 2024 and February 2025), in relation to the MAC for water classes I and II [11].

The correlation plots and results from the analyzed water samples were thoroughly studied, and appropriate interpretations were provided for them.

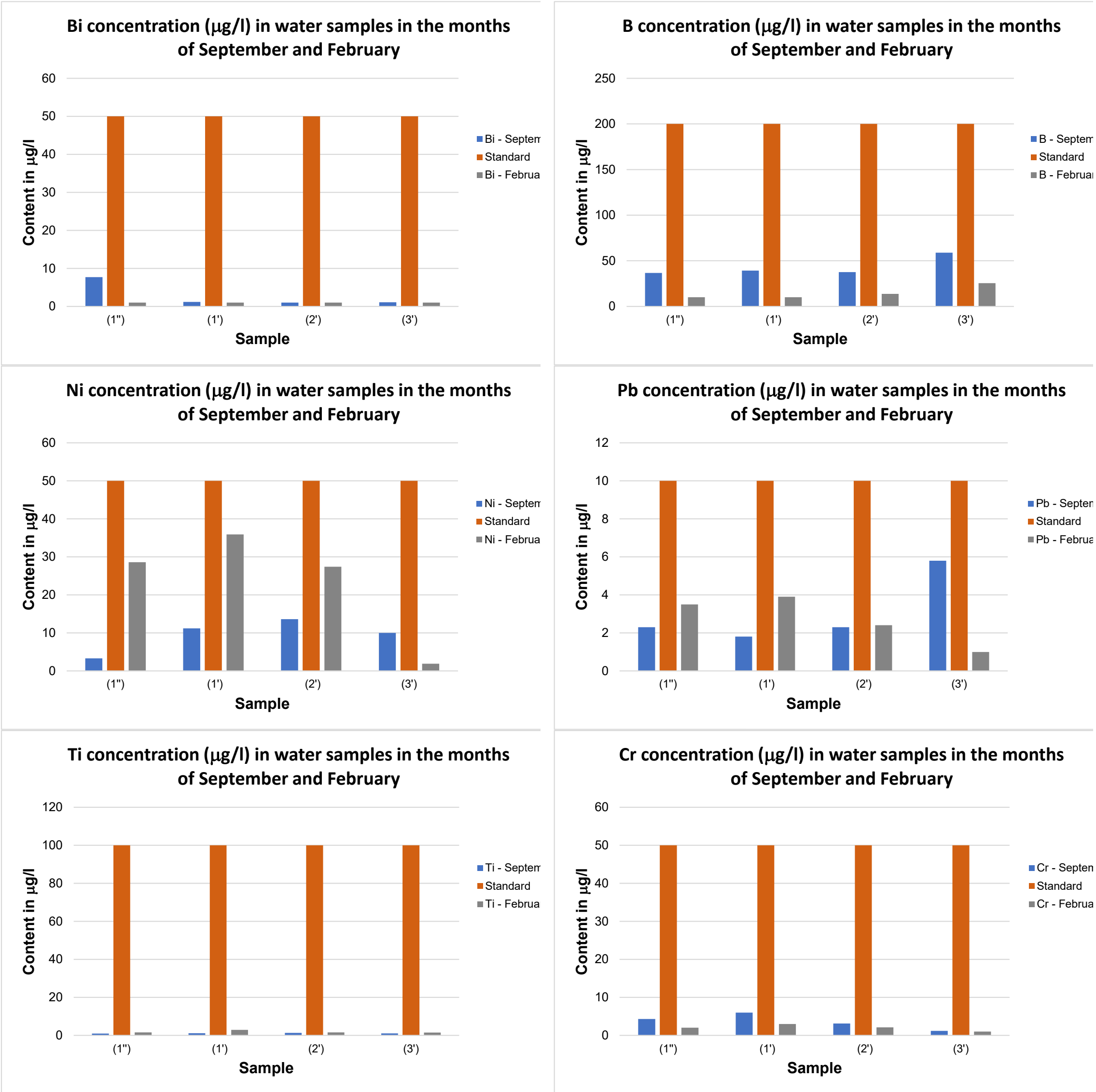



Figure 6. Concentration of elements in water samples within the limits of MAC for water classes I and II (September and February 2024/25), expressed in μg/l

From the correlation diagrams in Figure 6, it can be concluded that the concentrations of these elements in the water are within the limits of the allowed concentrations, i.e., they are below the limits for allowed concentrations in both examined time periods.

Additionally, from the concentration diagrams, it can be observed that although the concentrations of the elements are within the maximum allowed concentration (MAC), they vary differently between the two examined time periods. Specifically, certain metal concentrations are more pronounced in September, while others are more pronounced in February. This is the result of their different solubility, migratory behavior, and leaching during the different time periods of examination.

The concentrations of As, Cu, Co, Ag, and Ti in the tested water samples are variable, occasionally increased depending on the location and the testing period (Figure 7).

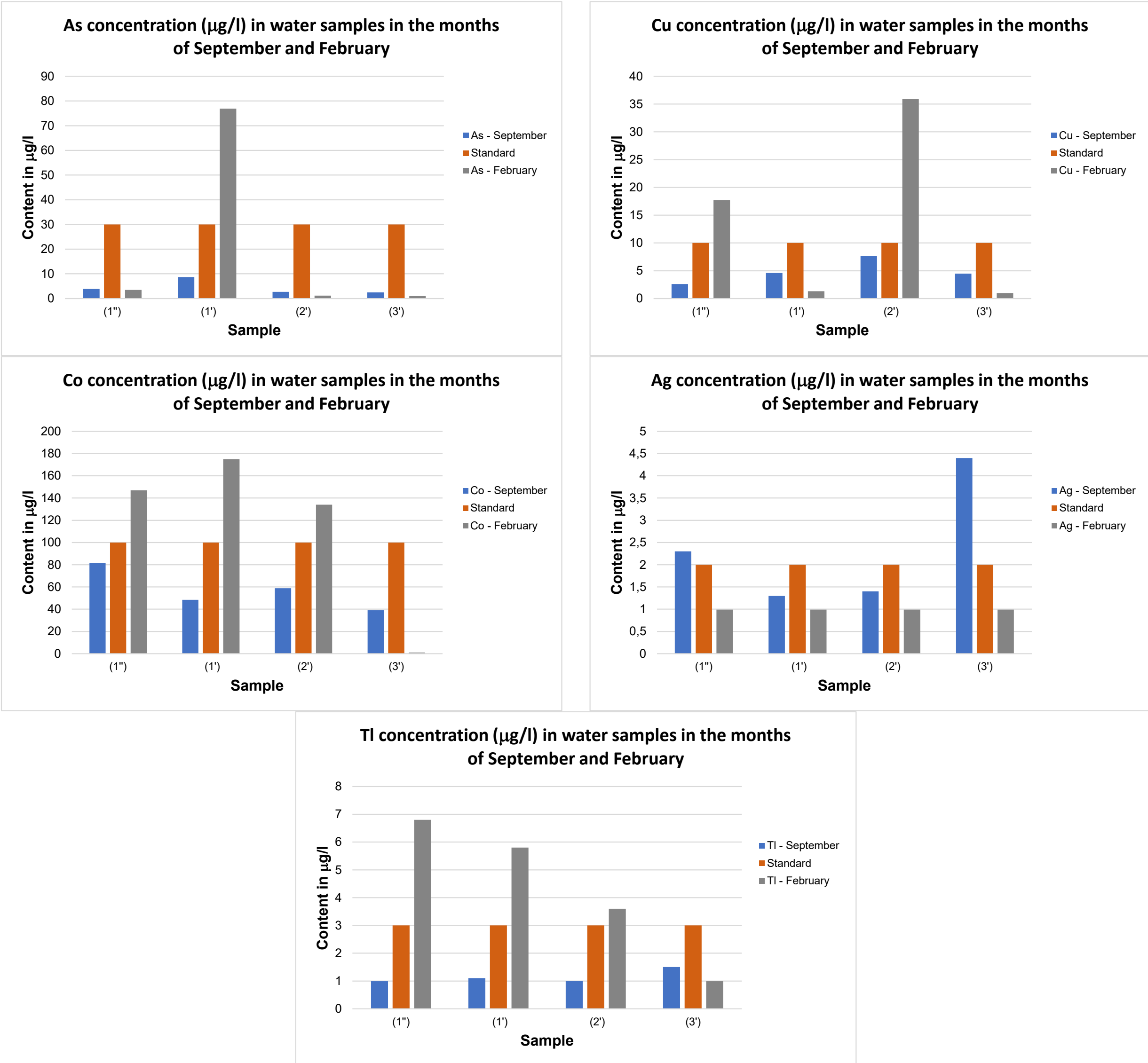



Figure 7. Concentration of As, Cu, Co, Ag, and Tl in water samples in relation to the MAC for water classes I and II (September and February 2024/25), expressed in μg/l

From the concentration diagrams in Figure 7, it can be concluded that the concentrations of As, Cu, Co, and Tl are within the MAC limits in September, while in February the concentrations in the water samples from the Plesenska River and the tested faucet have shown increased values. Also, As is increased only in the water sample from the Plesenska River above the village (1'), and Cu is increased only in the water samples from the faucet (1'') and from the Plesenska River below the village (2'). It is also observed that for all elements, except for Ag, the increased concentrations are found in February, while for Ag, the increased concentrations are in September, and only in the water from the faucet (1'') and from the confluence of the Kiselička River into the Zletovska River (3').

Increased concentrations in the water samples compared to the maximum allowed concentrations (MAC) were found for Fe, Cd, Mn, and Zn (Figure 8).

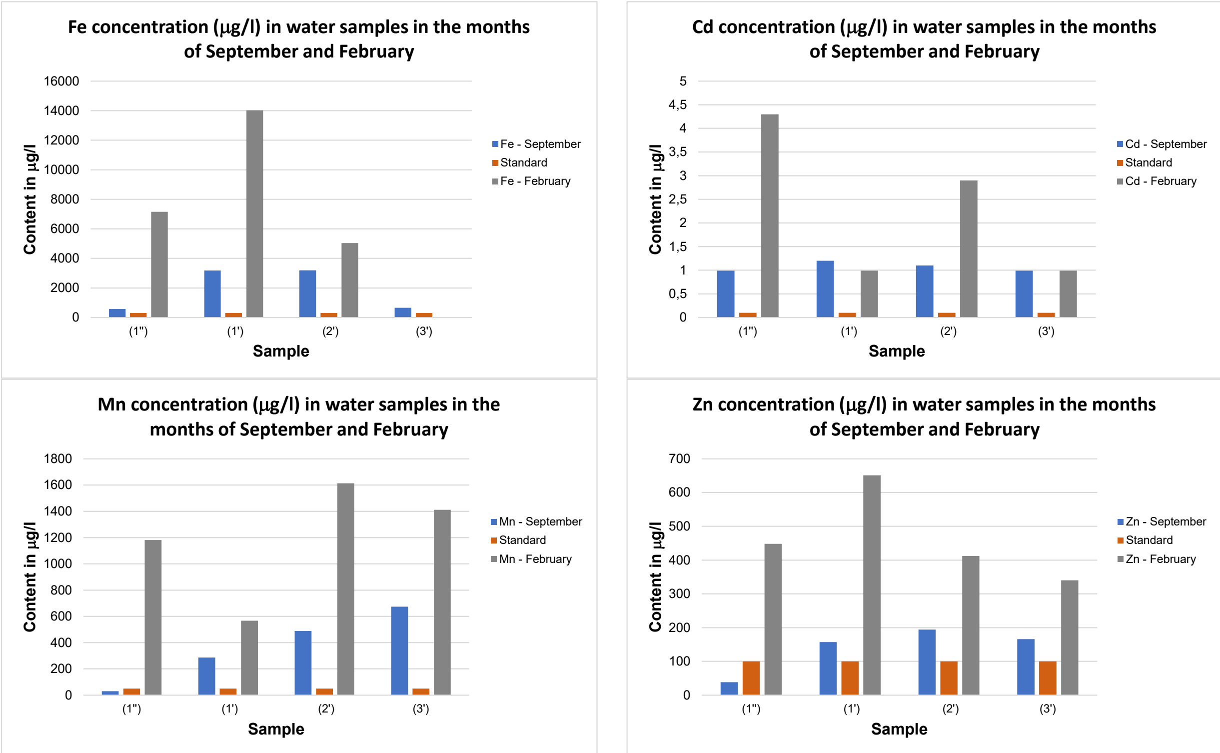
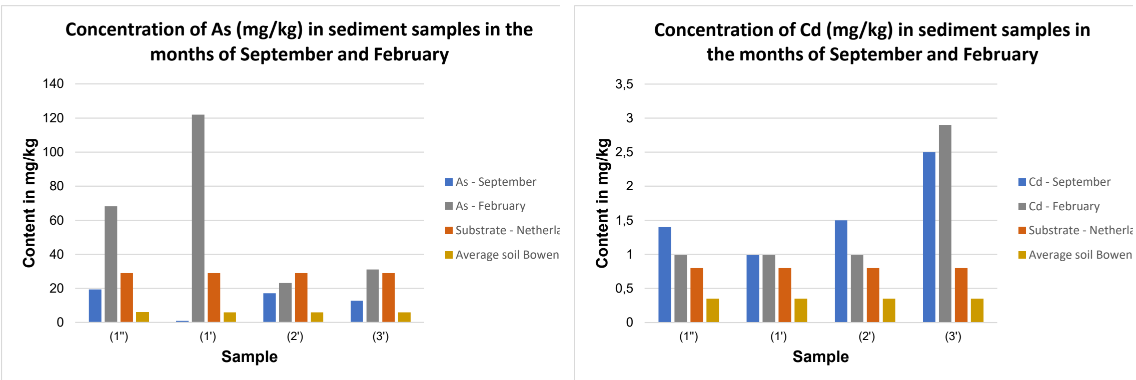



Figure 8. Elements with increased concentrations in water samples in relation to the MAC for water classes I and II (September and February 2024/25), expressed in µg/l

From the concentration diagrams in Figure 8, it can be observed that the concentrations of all elements, except for some samples (Mn and Zn in the sample from the faucet – 1''), are increased in both examined time periods. It is clearly evident that the increase in element concentrations is generally greater in February, as a result of higher leaching, solubility, and migration of the elements.

Elemental concentration diagrams were also constructed for the sediment samples (Figure 9) for the two studied time periods (September 2024 and February 2025) and in relation to the two reference values for the elements that were previously mentioned.

The concentration diagrams and the results from the analyzed sediment samples were thoroughly examined, and appropriate interpretations were provided.



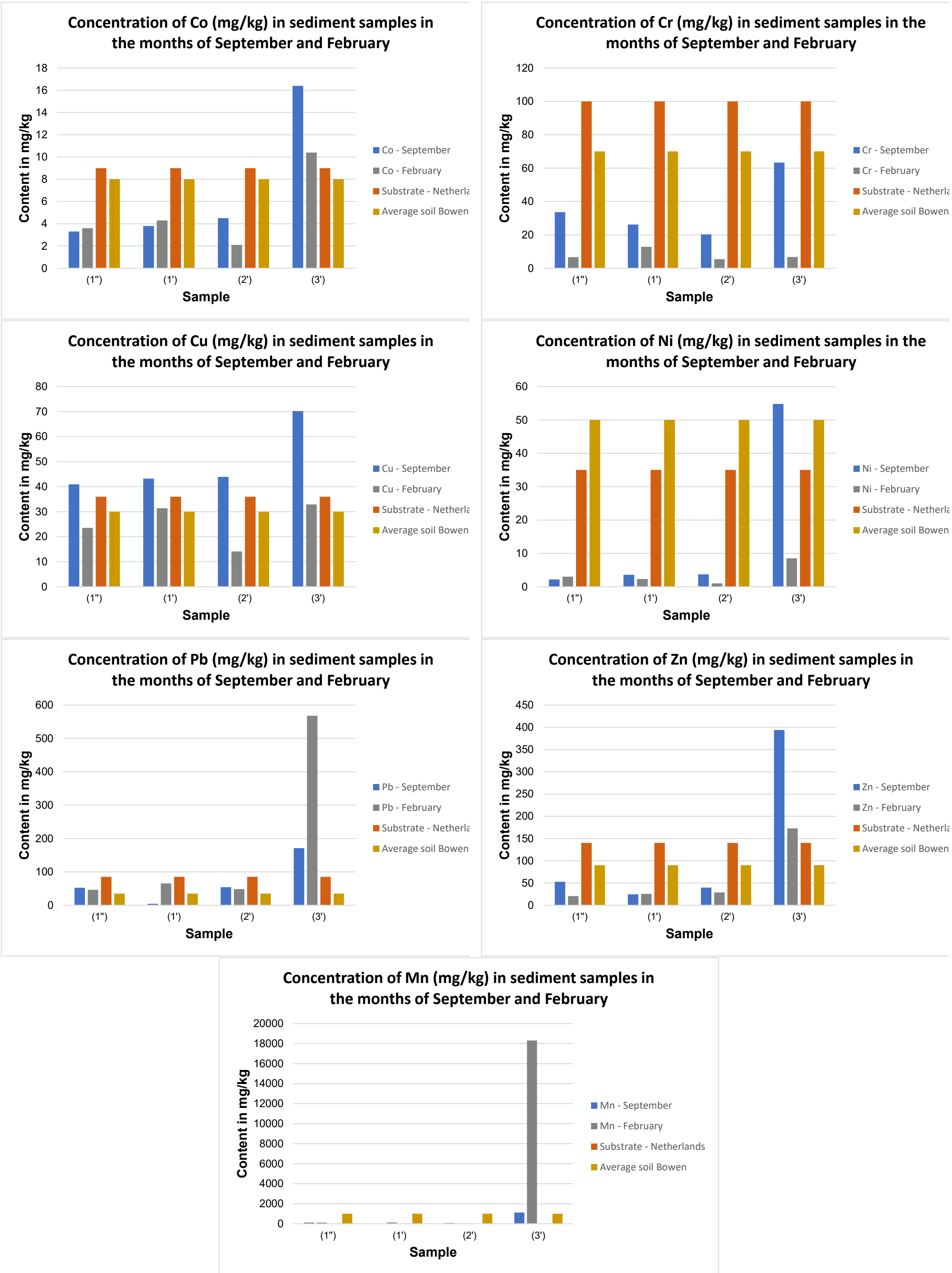



Figure 9. Concentration of individual elements in sediment samples (September and February 2024/25) according to the national substrate concentration for the Netherlands and Environmental Chemistry of the Elements [12], expressed in mg/kg

From the correlation diagrams in Figure 9, it can be concluded that the highest concentrations of elements in the sediments are present in the samples taken at the confluence of the Kiselica River into the Zletovska River - 3' (Cd, Co, Cu, Ni, Pb, Zn, and Mn). Their concentrations are variably increased in relation to the autumn and early spring periods. Pb, Zn, Ni, Co, and Mn (Mn-MDK only according to Bowen, 1979) show increased concentrations only in the samples taken from the sediments at the confluence of the Kiselica River into the Zletovska River (3'), according to both applied reference values. Their increased concentrations vary due to the degree of solubility, mobility, and leaching of the elements depending on the testing period.

The concentrations of Cr are within the maximum permissible concentrations according to both applied boundary values in both the autumn and early spring periods.

The concentrations of Cu in the sediment samples are increased in all samples only in September concerning both boundary values and slightly increased in some samples in February according to Bowen, while the concentrations of Cd are increased in all samples in both studied periods related to both applied boundary values. For both Cd and Cu, particularly increased concentrations are observed in the sediments taken at the confluence of the Kiselica River into the Zletovska River (3').

For As, it is important to mention that its concentrations in the sediments, when compared with the concentrations for As according to Bowen, are increased in all tested samples. When compared with the concentrations for As in the Netherlands substrate, they are increased only in February, and notably in the samples from the faucet (1") and from the Plesenska River above the village of Plesenci (1'), and slightly at the confluence of the Kiselica River into the Zletovska River (3').

## CONCLUSION

According to the analysis results of the water and sediments from the Plesenska River, the Kiselica River, and the faucet in the immediate vicinity of the Plesenska River, it can be concluded that they are characterized by increased concentrations of certain elements, or heavy metals. Their presence in the waters and sediments is a result of both natural and anthropogenic influences and contamination. Natural contamination is indicated by the results from the waters and sediments of Plesenska River and the tested faucet, while anthropogenic contamination is suggested by the results from the confluence of the Kiselica River into the Zletovska River.

Of particular interest are the increased concentrations of certain heavy metals, especially Cd in the water from the faucet, which is used by the local population as "holy water." Therefore, for safety and health reasons, it is recommended that this water should not be consumed by people, nor used for watering, watering livestock or any other purposes.

Furthermore, the application of the reference values for elements from the National Substrate Concentration for the Netherlands and Environmental Chemistry of the Elements [12] indicates contrasting differences in the reference values for some elements (e.g., As), meaning that using these values can lead to different results for the same element, depending on whether its concentrations are increased or within the reference values.

In this context, due to the absence of legislative regulation for soils and sediments in our country with clearly defined boundary values for harmful and dangerous substances in them, it is recommended and emphasized that efforts should be made as soon as possible to adopt and enforce such legislation.

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