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Concentration of Heavy Metals in Sediment and Water of Përlepnica Lake, Kosovo

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Abstract
Gjilan is a city located in southeast Kosovo. The main supplier of drinking water for the city is the Përlepnica Lake where heavy metals in sediment and water, and physicochemical parameters were analysed. Average concentrations of metals in soil sediment were found in the following order: Mn (708.65 mg kg^{-1}) > Zn (604.55 mg kg^{-1}) > Pb (264.26 mg kg^{-1}) > Cr (71.43 mg kg^{-1}) > Cu (61.08 mg kg^{-1}) > Ni (37.27 mg kg^{-1}) > Cd (6.32 mg kg^{-1}) > Fe (1.83 mg kg^{-1}). Average concentrations of metals in water were found in the following order: Fe (0.295 mg l^{-1}) > Zn (0.066 mg l^{-1}) > Cu (0.020 mg l^{-1}) > Cr (0.035 mg l^{-1}) > Cd (0.003 mg l^{-1}) > Mn (0.003 mg l^{-1}). The concentrations of heavy metals in sediment, such as Cd, Cu, Mn, Pb, and Zn, were not in accordance with the "New Dutch List" (soil and ground water criteria used in The Netherlands for contaminated land), and the concentration of Fe and Cu in water was not in accordance with EU Directive 75/440.

Keywords
Heavy metals, Përlepnica Lake, water, sediment, pollution, ICP-OES, AAS

1 Introduction

Gjilan is one of the seven largest cities in Kosovo.[1] Most of the municipality uses groundwater as drinking water[2] and the other half of the citizens in this municipality are supplied by Lake Përlepnica.

The quality of sediments influences the water quality in the aquatic environment.[3] The behaviour of metals in natural waters is determined by the water chemistry and sediment composition.[4] The total heavy metal content in the sediment, related to toxicity and mobility, provides incomplete information, but can be considered as pollution indicator.[5] Heavy metals (HMs) are one of the most serious environmental pollutants due to their toxic effects. They are persistent and abundant, and can accumulate in aquatic ecosystems.[6]

Monitoring and assessment of water quality is a crucial sustainability issue for surface waters.[7,8] HMs pollution of drinking water is a critical issue affecting numerous countries worldwide. The reason for studying the Përlepnica Lake was to determine the concentration of heavy metals in the sediment, and whether the HMs, present in the sediment, can penetrate the lake water. During the study, chemical analyses expressed high concentration of HMs in the sediment as well as the penetration of the heavy metals into the water. In addition, the physicochemical parameters of the lake were analysed which, in some samples, had a change in concentrations compared to the allowed concentrations for water quality. This work aimed to determine the heavy metals in water, sediment, and physicochemical parameters of Përlepnica Lake (Fig. 1). The following HMs were determined in the water of Përlepnica Lake: Fe > Zn > Cu > Cr > Cd > Mn, while the following

Fig. 1 – Përlepnica Lake (published with author's permission: Arben Llapashtica, 2020)
Slika 1 – Jezero Përlepnica (objavljeno uz dopuštenje autora fotografije: Arben Llapashtica, 2020.)

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HMs were determined in the sedimentation of the lake: Mn > Zn > Pb > Cr > C u > Ni > Cd > Fe. The conducted analysis of the water of Përlepnica Lake showed the presence of toxic metals (Fe, Zn, and Cu) that were not in accordance with EU Directive 75/440, while in the sediment of Përlepnica Lake there was a high concentration of the following HMs: Fe, Pb, Cd, Cu, Mn, and Zn. Based on these data, this study focused on the analysis of sediment and water in Përlepnica Lake, and the main objective of this study was to assess the quality of water and sediment as well as to show the levels of heavy metals in the water and sediments.

2 Location of sample collection

Përlepnica Lake is a small lake in the western part of Kosovo, surrounded by the Gollak mountains where the water springs. It lies northeast of the city of Gjilan from which water is supplied. A branch of Morava e Binçës (South Morava) flows through this lake, which is otherwise considered to be the largest lake in eastern Kosovo. Përlepnica Lake was built in 1966, and it is used for drinking as well as irrigation of 350 ha of arable land. The accumulation of Përlepnica Lake is formed in the river of the same name. Its water volume is 4.2 million m^3. It has an area of 1.634 km^2 and a flow of 16.32 m^3 of water *per* second. Fig. 2 shows the water and sediment, and water sampling points.

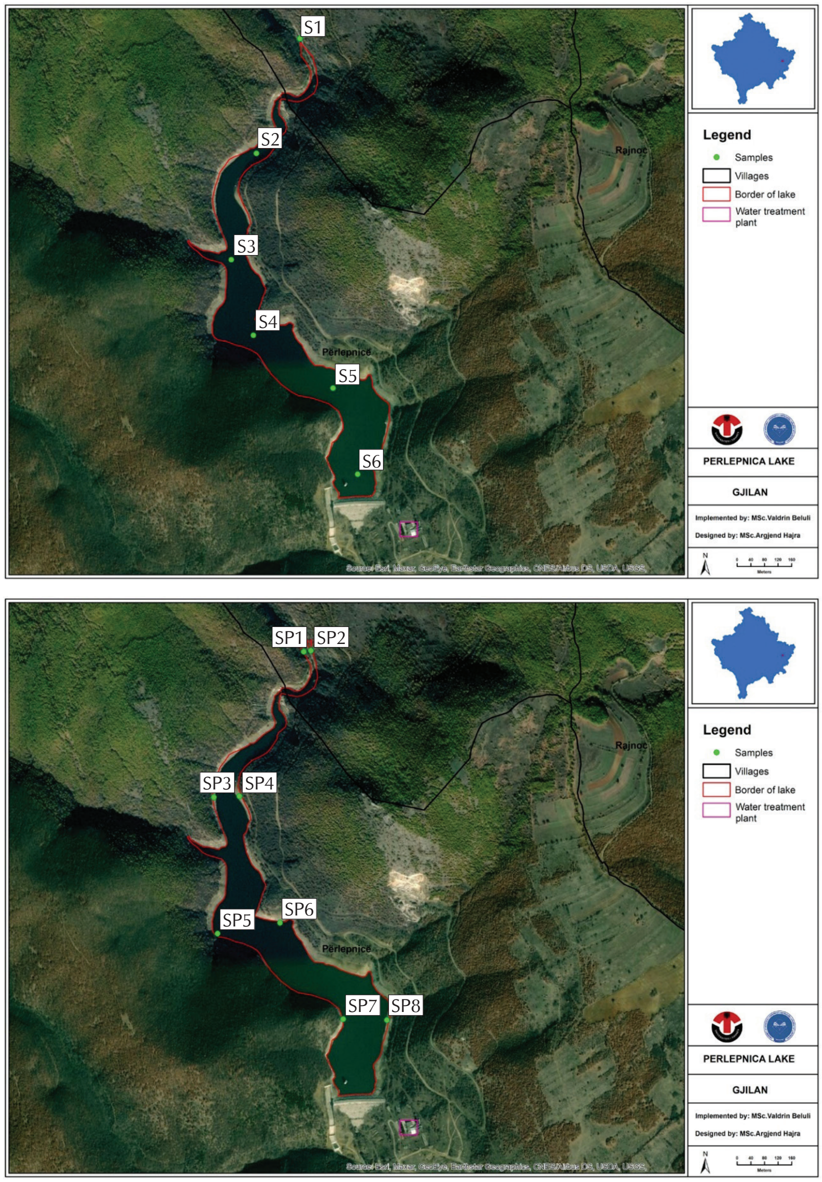

Fig. 2 – Water sampling (SP$_1$–SP$_6$); sediment sampling (SP$_1$–SP$_8$) in Përlepnica Lake

Slika 2 – Uzorkovanje vode (SP$_1$–SP$_6$); uzorkovanje sedimenta (SP$_1$–SP$_8$) u jezeru Përlepnica

2.1 Atmospheric conditions in the village of Përlepnica

Kosovo has quite different climates in the four seasons of the year. According to the data of the Hydrometeorological Institute in Kosovo, temperatures during the summer season reach an average of 35 °C, while temperatures during the winter average reach −15 °C. The summer season in the city of Gjilan is quite dry and in the absence of atmospheric rain (Fig. 3). The city of Gjilan has only one artificial lake, the Përlepnica Lake. The water in the lake is still used for drinking, but this lake dries almost completely during the summer, because the water requirements consume almost 90 % of the water in the lake. The predominant agricultural land of the Përlepnica village is cultivated with various fruits and vegetables, as the land in this part is very fertile and suitable for cultivation.

Fig. 3 – Drought in Përlepnica Lake in 2020 (published with author's permission: Gentian Syla)

Slika 3 – Suša jezera Përlepnica 2020. (objavljeno uz dopuštenje autora fotografije Gentian Syla)

2.2 Sampling

According to the standard methods (APHA/AWWA/WEF, 2012),[9] the water samples were collected and transported to the laboratories[10,11] (Fig. 4). Samples intended for chemical analysis had to be collected during normal operating hours, 15 to 30 cm below the surface of the water,[12] and the volume of the water samples was 1 dm^3. A day before use, all glassware was washed with 1–2 % HCl solution, rinsed with distilled water, and oven-dried.[7]

Water samples were taken at three different times in 2020 (March, April, June), while sediment samples were taken in January 2020. The weight of the sediment samples was approximately 1 kg. Sediment samples were collected by hand with a stainless-steel shovel at sampling depth of 30 cm. The sediment samples were wet, and, before being analysed, the samples were oven-dried at 105 °C. Water and sediment samples were stored in the laboratory at 20 °C until usage.[13]

Samples were taken at the coordinates presented in Tables 1 and 2.

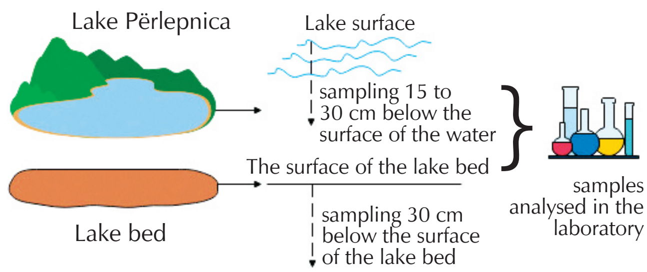

Fig. 4 – Sampling of sediments in the bed and water of Përlepnica Lake
Slika 4 – Uzorkovanje sedimenata u koritu i vodi jezera Përlepnica

2.3 Conservation and transportation of water samples

Methods Standard ISO 9001: Hydrometeorology, Institute of Kosovo[14] gave some recommendations for the conservation of the samples for the analysis. That means that the samples are immediately placed in the refrigerated portable shaved to certain conditions. For physicochemical analysis, if the samples are not conserved, the time between sampling and performing analyses should be much shorter. For the determination of heavy metals, 100 ml of each sample was taken separately and then conserved by treatment with 0.5 ml of concentrated HNO_3.

Table 1 – Coordinates of sediment sampling in Përlepnica Lake
Tablica 1 – Koordinate uzorkovanja sedimenata u jezeru Përlepnica

Samples	Coordinate X	Coordinate Y
SP_1	42°32′02.6″N	21°30′51.4″E
SP_2	42°32′02.7″N	21°30′52.3″E
SP_3	42°31′48.9″N	21°30′39.7″E
SP_4	42°31′49.0″N	21°30′43.0″E
SP_5	42°31′35.8″N	21°30′39.0″E
SP_6	42°31′37.0″N	21°30′48.1″E
SP_7	42°31′27.9″N	21°30′56.2″E
SP_8	42°31′27.7″N	21°31′02.3″E

* Explanations: SP_1–SP_8 = sampling points (Fig. 2)

3 Experimental

3.1 Chemical analysis

Samples were taken (SP_1–SP_8) at the bottom of the lake for the analysis of HMs with inductively coupled plasma-optical emission spectroscopy (ICP-OES). Samples of water (SP_1–SP_6) were also taken for the determination of HMs with atomic absorption spectrometry (AAS). In addition, several physicochemical parameters in the Përlepnica Lake were determined, such as temperature (°C), odour, pH, nephelometric turbidity unit (NTU), conductivity of water (*CW*), dissolved oxygen (DO), alkalinity (A-HCl), bicarbonates (HCO_3^-), general hardness (*GH*), calcium (Ca^{2+}), magnesium (Mg^{2+}), chlorides (Cl^-), nitrites (NO_2^-), nitrates (NO_3^-), and sulphates (SO_4^{2-}).

3.2 Determination of heavy metals in water and sediments

AAS is one of the most used methods in analytical practice. It is used for the quantitative analysis of most metals in the periodic table of elements, and approximately 70 elements can be defined.[15]

The AAS instrument defined the following HMs in the water: Mn, Zn, Pb, Cr, Ni, Cd, Fe, Hg, and Cu.

Chemical analysis of HMs in sediment was performed in the commercial laboratory "Agrovet" using Perkin Elmer "Optima 2100 DV ICP Optical Emission Spectrometer".[16] The ICP-OES (Method: ISO 12914, EPA 6010C) instrument defined the following HMs in the sediment: Mn, Zn, Pb, Cr, Ni, Cd, Fe, Hg, and Cu.

The HACH® Model DR/2010 Spectrophotometer is a microprocessor-controlled single beam instrument for color-

Table 2 – Coordinates of water sampling in Përlepnica Lake
Tablica 2 – Koordinate uzorkovanja vode u jezeru Përlepnica

Samples	Coordinate X	Coordinate Y
SP_1	42°32′04.4″N	21°30′50.9″E
SP_2	42°31′53.5″N	21°30′45.2″E
SP_3	42°31′43.4″N	21°30′41.9″E
SP_4	42°31′36.2″N	21°30′44.7″E
SP_5	42°31′31.1″N	21°30′54.9″E
SP_6	42°31′22.9″N	21°30′58.0″E

* Explanations: SP_1–SP_6 = sampling points (Fig. 2)

imetric testing in the laboratory or in the field.[17] The pH was determined using a portable multi-parameter analyser, WTW 3010. The conductivity of water (*CW*), also known as specific conductivity, represents the ability of water to convey electricity and is related to the concentration of ionized substances in water. *CW* was determined with WTW Cond 3110, *DO* was determined with WTW Oxi 315i, and NTU with 2100N ISC Turbidimeter (ISO Method 7027).[1]

Sulphate (SO_4^{2-}) concentration was determined using Sulfa Ver 4 (0–70 $mg\,l^{-1}$), method 8051, and the absorbance level was then measured using a spectrophotometer (HACH® DR/2010) at λ = 450 nm. Nitrite (NO_2-N) concentration was determined using Nitri Ver 3 reagent (test 0–0.300 $mg\,l^{-1}$, method 8507), and the absorbance level was measured using a spectrophotometer (HACH® DR/2010) at λ = 507 nm.[17]

General hardness (*GH*) was analysed by adding 2–5 ml of buffer and indicator (black erythromycin) in very small quantities to a sample of 100 ml of water. Following the addition of the indicator, the solution became red or light red, and the titration was done with complexon III or ethylenediaminetetraacetic acid (EDTA) until the solution changed its colour to intensive blue.[1] The calculation was based on Eq. (1).

$$GH(°dH) = \frac{V_{EDTA} \cdot c_{EDTA} \cdot 56 \cdot 1000}{V_s} \quad (1)$$

V_{EDTA} is titration volume of EDTA (ml), c_{EDTA} is the EDTA concentration (0.01 $mol\,l^{-1}$), and V_s is the volume of the used sample (ml).

Carbonate strength (*CS*) is defined as the alkalinity of methyl-orange. A volume of 100 ml water sample was transferred to 500-ml Erlenmeyer flask and 2–3 drops of methylene chloride were added. The titration was performed with standard solution HCl (c = 0.01 $mol\,l^{-1}$) until the colour changed to orange.[1] The analysis results were calculated in the German degrees (°dH) of water hardness according to Eq. (2).

$$CS(°dH) = 2.8 \cdot V_{HCl} \cdot c_{HCl} \quad (2)$$

V_{HCl} is consumed volume of HCl solution in concentration c_{HCl} = 0.01 $mol\,l^{-1}$.

Calcium (Ca^{2+}) or CaO equivalent can be determined by titration with standard EDTA solution in the presence of murexide (ammonium purpurate). First, 5 ml of NaOH in concentration 2 $mol\,l^{-1}$ was added to 100 ml of water sample and the sample was titrated with EDTA until the colour changed from red to purple. The determination of Ca^{2+} (in $mg\,l^{-1}$) was calculated by Eq. (3).[11]

$$Ca^{2+}\ (mg\ l^{-1}) = \frac{V_{EDTA} \cdot c_{EDTA} \cdot 56 \cdot 1000}{V_s} \quad (3)$$

V_{EDTA} is volume (ml) of EDTA solution in concentration c_{EDTA} = 0.01 $mol\,l^{-1}$ and V_s is the volume of sample used, M_{CaO} = 56 $g\,mol^{-1}$. The content of Mg^{2+} or MgO equivalent ($mg\,l^{-1}$) was calculated using Eq. (4).[1]

$$Mg^{2+}\ (mg\ l^{-1}) = \frac{GH / °\,dH - Ca^{2+}\ (mg\ l^{-1})}{10} \cdot 7.19 \quad (4)$$

Water sample alkalinity (A-HCl) is the measurement of its capacity to neutralize acids. Four drops of phenolphthalein were added to 100 ml of the sample. If the 100-ml solution becomes purple, the pH of water contained bases is above 8.30, and if the solution does not turn purple, 2–3 drops of methyl-orange are added, which turns the solution yellow. The solution was then titrated with HCl (c = 0.01 $mol\,l^{-1}$) until it turned orange, and the amount of titre used was recorded.

Determination of chlorides (Cl^-) was carried out in an Erlenmeyer flask containing 100 ml of water sample (adjust pH to 7–10 if necessary). With the addition of 1 ml of K_2CrO_4 0.257 $mol\,l^{-1}$, the sample turned yellowish. Titration was done with silver nitrate $AgNO_3$ (c = 0.01 $mol\,l^{-1}$), and stopped when the solution turned light red.[1] The value of chlorides ($mg\,l^{-1}$) in the sample was calculated according to Eq. (5).

$$Cl^-(mg\ l^{-1}) = 35.453 \cdot c \cdot M \cdot \frac{(V_1 - V_2)}{V_s} \quad (5)$$

V_1 is volume of titre for sample (ml), V_2 is volume of titre for blind sample (ml), c= 0.01 $mol\,l^{-1}$ is molar concentration of $AgNO_3$, and V_s is the volume of sample used (100 ml in our research).

4 Results and discussion

4.1 Physicochemical parameters in the water of Përlepnica Lake

Water quality assessment in lakes is a widely recognized problem. In order to understand water quality conditions, monitoring the physicochemical parameters of lake water is crucial, because it provides essential information for efficient water management practices.[18]

The water of Përlepnica Lake has no odour and no taste, which was determined during sampling, because this lake has no industry or urban development to cause water pollution.

The temperature of surface waters is influenced by latitude, altitude, season, time of day, air circulation, cloud cover, the flow, and the depth of the water. Water temperature varies due to atmospheric conditions during the seasons.[11] Water temperature is a good indicator of whether water is polluted or not. The acceptable limit of water temperature according to Directive 75/440 BE is 22 °C. The temperature values of all the water samples were found to be in the range between 17.5 and 18.8 °C, the average temperature value was 18.06 °C, and the samples analysed were in accordance with Directive 75/440 BE (Table 3). The pH is classed as one of the most important water quality parameters. Measurement of pH relates to the acidity or alkalinity of the water. A sample is considered to be acidic at pH below 7.00, and alkaline at pH higher than 7.0. Acidic water can lead to corrosion of metal pipes and plumping

Table 3 – Physicochemical parameters of the Përlepnica Lake (March 2020)
Tablica 3 – Fizikalno-kemijski parametri jezera Përlepnica (ožujak 2020.)

Parameters and symbols	Unit	Directive 75/440 BE	SP_1	SP_2	SP_3	SP_4	SP_5	SP_6	Average
temperature	°C	22	18	17.50	18.10	18.80	17.80	18.20	18.06
odour	–	no	no	no	no	no	no	no	no
taste	–	no	no	no	no	no	no	no	no
nephelometric turbidity unit (NTU)	NTU	before processing <5	5.40	5.20	5.40	4.40	5.70	5.25	5.22
pH	pH	6.50–8.50	7.28	8.25	8.00	8.10	8.01	8.20	7.97
chlorides (Cl^-)	mg l^{-1}	200	180	185	182	180	181	184	182
nitrites (NO_2^-)	mg l^{-1}	0.005	0.003	0.003	0.003	0.002	0.002	0.002	0.0025
nitrates (NO_3^-)	mg l^{-1}	25	5.10	5.20	4.50	5.10	4.21	6.30	5.06
conductivity of water (CW)	µS cm^{-1}	200-800	390	382	395	384	400	406	392.83
sulphates (SO_4^{2-})	mg l^{-1}	150	39.04	39.68	39.04	40.00	41.62	41.60	40.16
calcium (Ca^{2+})	mg l^{-1}	<200	63.20	64.12	59.31	55.31	61.70	64.00	61.27
magnesium (Mg^{2+})	mg l^{-1}	<50	8.50	8.41	12.51	11.76	9.57	12.1	10.47
alkalinity (A-HCl)	ml	10.50	3.10	3.10	3.20	2.90	3.01	2.80	3.01
general hardness (GH)	°dH	30	10.93	10.92	11.20	10.47	10.20	11.30	10.83
bicarbonates (HCO_3^-)	mg l^{-1}	630	189.10	189.10	195.20	176.90	179.40	180	184.95
carbon strength (CS)	°dH	/	8.68	8.68	8.96	8.12	8.80	8.90	8.69
dissolved oxygen (DO)	mg l^{-1}	>5	3.69	3.80	4.60	4.90	4.80	5.10	4.35

systems.[19] The normal water pH range mentioned in Directive 75/440 BE and guidelines is between 6.50 and 8.50. The pH values of all the water samples were found to be in the range between 7.28 and 8.25, the average pH value was 7.97, and the samples analysed were in accordance with Directive 75/440 BE (Table 3). Electrical conductivity is the ability of any medium, water in this case, to carry an electric current. The presence of dissolved solids such as calcium, chloride, and magnesium in water samples carries the electric current through water.[19] The measured *CW* values of all the samples are plotted in Table 3. According to Directive 75/440 BE, the maximum allowable level of conductivity is (200–800) µS cm^{-1}. The results showed that the measured conductivity of all water samples ranged from 382 to 406 µS cm^{-1}, and the average conductivity value was 392.83 µS cm^{-1} (Table 3). High conductivity may lead to lowering the aesthetic value of the water by giving mineral taste to the water. For industrial and agricultural activity, monitoring of the water conductivity is crucial. Water with high conductivity may cause corrosion of the metal surface of equipment such as boiler.[20] Turbidity is the cloudiness of water caused by a variety of particles, and is another key parameter in drinking water analysis.[19] The turbidity (NTU) results for all water samples studied are shown in Table 3. According to Directive 75/440 BE, the maximum allowable level of NTU is < 5. The lowest turbidity value was 4.40 NTU, the highest value was 5.40 NTU, and the average NTU value was 5.22. High NTU value was present in samples SP_1, SP_2, SP_3, SP_5, and SP_6, while only sample SP_4 was in accordance with the Directive 75/440 BE (Table 3). NTU is usually very high in Përlepnica Lake in the winter season when rainfall is more frequent.

Dissolved oxygen (DO) is considered an important measure of water quality as it is a direct indicator of an aquatic resource's ability to support aquatic life.[21] According to Directive 75/440 BE, the maximum allowable level of DO is > 5 mg l^{-1}. The lowest DO value was 3.69 mg l^{-1}, the highest value was 5.10 mg l^{-1}, and the average DO value was 4.35 mg l^{-1} (Table 3). Based on the analysed samples, DO was not in accordance with Directive 75/440 BE, and the results of DO showed that the oxygen in the water of Përlepnica Lake was consumed by organic matter that decomposed in the water. Alkalinity (A-HCl) refers to the capability of water to neutralize the acid.[22] The acceptable limit of A-HCl in water according to Directive 75/440 BE is 10.50 ml. The results showed that the measured A-HCl of all water samples ranged from 2.80 to 3.20 ml, and the average A-HCl value was 3.01 ml (Table 3). The value of alkalinity in water depends on the presence of bicarbonate and carbonates.[23] The low alkalinity of water in the Përlepnica Lake was due to its low concentration of bicarbonates (HCO_3^-). The acceptable limit of HCO_3^- in water according to Directive 75/440 BE is 630 mg l^{-1}. The results showed that the measured conductivity of all water samples ranged from 176.90 to 195.20 mg l^{-1}, and the average HCO_3^- value was 184.95 mg l^{-1} (Table 3). General hardness (*GH*) is mainly contributed by bicarbonates, carbonates, sulphates, and chlorides of calcium and magnesium. Therefore, the principal hardness-causing ions are calcium and magnesium.[24] According to Directive 75/440 BE, the maximum allowable level of GH/°dH is 30 °dH. The lowest GH/°dH value was 10.2 °dH, the highest value was 11.3 °dH, and the average GH/°dH value was 10.83 °dH (Table 3). Based on the sample values, the water of Përlepnica Lake is of medium strength °dH. The reason why the water of Përlepnica Lake has medium hardness °dH is due to the low concentration of calcium and magnesium. The calcium (Ca^{2+}) results for all studied water samples of Përlepnica Lake are shown in (Table 3). According to Directive 75/440 BE, the maximum allowable level of Ca^{2+} is < 200 mg l^{-1}. The lowest Ca^{2+} value was 55.31 mg l^{-1}, the highest value was 64.12 mg l^{-1}, and the average of Ca^{2+} was 61.27 mg l^{-1} (Table 3). As mentioned previously, magnesium (Mg^{+2}) is a contributor to the general hardness of water. The acceptable limit of Mg^{+2} in water according to Directive 75/440 BE is < 50 mg l^{-1}. The results showed that the measured Mg^{+2} of all water samples ranged from 8.41 to 12.10 mg l^{-1}, and the average Mg^{+2} value was 10.47 mg l^{-1} (Table 3). Chloride (Cl^-) is present in all-natural waters, and its content moves to a greater extent in natural waters.[25] Dissolved Cl^- in surface waters occurs naturally from the geology, but high concentrations typically result from runoff of de-icing salts.[26] According to Directive 75/440 BE, the maximum allowable level of Cl^- is 200 mg l^{-1}. The results showed that the measured Cl^- of all water samples ranged from 180 to 185 mg l^{-1}, and the average Cl^- value was 182 mg l^{-1} (Table 3). The high amount of chlorides also indicates an undesirable effect on the structure of metal pipes, causing a salty taste in the water.[23] Sulphates (SO_4^{3-}) are more commonly found in groundwater than in rivers and lakes.[26] The high concentration of SO_4^{3-} in water is caused by the oxidation of gypsum and the release of industrial wastewater.[24] According to Directive 75/440 BE, the maximum allowable level of SO_4^{3-} is 200 mg l^{-1}. The results showed that the measured SO_4^{3-} of all water samples ranged from 39.04 to 41.62 mg l^{-1}, and the average SO_4^{3-} value was 40.16 mg l^{-1} (Table 3). Based on the results of the analysed samples, the concentration of SO_4^{3-} was quite low compared to the value allowed by the Directive 75/440 BE. A high concentration of nitrate (NO_3^-) in water is not suitable for human consumption because it increases cancer risk.[27] NO_3^- in water mostly occurs when municipal or industrial waste is discharged or wastewater is released.[23] The acceptable limit of NO_3^- in water according to Directive 75/440 BE is 25 mg l^{-1}. The results showed that the measured NO_3^- of all water samples ranged from 5.10 to 6.30 mg l^{-1}, and the average NO_3^- value was 5.06 mg l^{-1} (Table 3). The value of NO_3^- in this lake is low because there is no pollution from solid waste or sewage. Nitrites (NO_2^-) can be in very high concentrations if the water has decomposing organic matter or if an organic industry releases its wastewater into a river or lake.[23] The acceptable limit of NO_2^- in water according to Directive 75/440 BE is 0.005 mg l^{-1}. The results showed that the measured NO_2^- of all water samples ranged from 0.002 to 0.003 mg l^{-1}, and the average NO_2^- value was 0.0025 mg l^{-1} (Table 3). The low nitrite concentrations in Përlepnica Lake are good result because this expresses inactivity of decomposition of organic matter in the water.

During the analysis of samples (SP_1–SP_6), all physicochemical parameters of the Përlepnica Lake were in accordance with the Directive 75/440, except NTU, which was slightly higher, and DO, which was less than the permitted concentration EC 74/440 (Table 3). Based on Table 4, it can be seen that Përlepnica Lake has higher values in physicochemical parameters compared to Skadar Lake.

Table 4 – Comparison of concentration of physicochemical parameters in the water of Përlepnica Lake and Skadar Lake
Tablica 4 – Usporedba koncentracija fizikalno-kemijski parametara u vodi jezera Përlepnica i Skadarskog jezera

Parameters and symbols	Unit	Përlepnica Lake min–max (Kosovo), results of this study	Lake Skadar min–max (Albania)[30]
temperature	°C	17.50–18.80	8.60–14.58
nephelometric turbidity unit (NTU)	NTU	4.40–5.40	0–2.68
pH	pH	7.28–8.25	7.64–8.47
chlorides (Cl^-)	mg l^{-1}	180–185	1.60–5.80
nitrites (NO_2^-)	mg l^{-1}	0.002–0.003	0.003–0.03
nitrates (NO_3^-)	mg l^{-1}	5.10–6.30	0.004–3.37
conductivity of water (CW)	µS cm^{-1}	382–406	195–273
sulphates (SO_4^{2-})	mg l^{-1}	39.04–41.62	1.00–20.30
calcium (Ca^{2+})	mg l^{-1}	55.31–64.12	24.60–62.90
magnesium (Mg^{2+})	mg l^{-1}	8.41–12.10	5.10–5.13
general hardness (GH)	°dH	10.20–11.30	5.20–10.00
bicarbonates (HCO_3^-)	mg l^{-1}	176.90–195.20	129–226
dissolved oxygen (DO)	mg l^{-1}	3.69–5.10	7.90–13.10

4.2 Metal concentration in sediment

Heavy metal pollution has been widely studied because it causes environmental and public health problems worldwide.[29] Among chemical pollutants, heavy metals are of great concern because they are known to inflict several health disorders in humans.[30]

Iron (Fe) can initiate cancer mainly by the process of oxidation of DNA molecules.[31] Concentrations of Fe in all samples are very high (1.69–1.96 mg kg^{-1}; Table 5) and the samples are included in a single group .

However, according to "The Dutch List", Fe has no criterion value because it is a massive element in sediments. The average concentration of Fe in Përlepnica Lake (Kosovo) was 1.83 mg kg^{-1}, Badovc Lake had very high concentration of 19084 mg kg^{-1}, while no Fe was present in Balaton Lake and Skadar Lake (Table 6).

Table 5 – Concentration of heavy metals in the sediment of Përlepnica Lake (January 2020)
Tablica 5 – Koncentracija teških metala u sedimentu jezera Përlepnica (siječanj 2020.)

Samples	Unit	Metals								
		Fe	Cd	Cr	Cu	Mn	Ni	Pb	Zn	Hg
SP_1	mg kg^{-1}	1.79	8.28	77.31	61.66	930.92	38.84	324.00	763.47	*bdl
SP_2		1.96	4.31	71.35	63.54	664.34	34.06	172.12	430.21	*bdl
SP_3		1.69	6.93	62.08	52.31	621.85	31.46	260.56	658.93	*bdl
SP_4		1.84	10.40	65.98	63.97	897.81	35.68	346.60	927.76	*bdl
SP_5		1.95	6.05	74.33	57.29	505.56	38.54	274.89	728.75	*bdl
SP_6		1.86	6.82	65.07	89.99	719.55	32.35	325.60	707.29	*bdl
SP_7		1.76	3.73	71.88	40.24	648.89	44.62	123.38	319.38	*bdl
SP_8		1.80	4.10	83.50	59.65	680.34	42.62	286.98	300.68	*bdl
Average		1.83	6.32	71.43	61.081	708.65	37.27	264.26	604.55	*bdl
Standard Deviation (SD)		0.091	2.18	7.03	14.03	141.63	4.74	78.45	227.63	-
* Below the detection level (bdl)										
A	mg kg^{-1}	/	1	100	50	/	50	50	200	0.50
B		/	5	250	100	/	100	150	500	2
C		/	20	800	500	/	500	600	3000	10
Soil criteria used in the Netherlands for contaminated land (Dutch Target and Intervention Values, 2000 (the New Dutch List), Version, February 4, 2000). A simplified explanation of the ABC levels: A – level implies unpolluted, B – level implies pollution present and further investigation required, C – level implies significant pollution present and clean-up										
From the reanalysis of the samples, the change in concentrations ranged from 0.0001 to 0.0002 mg kg^{-1}										
Method: ISO 12914 EPA 6010 C										
The samples were wet and dried immediately in the laboratory at 105 °C.										

Cadmium (Cd) has toxic effects on the reproductive system, fertility, causes kidney damage, renal disorder, and is carcinogenic.[32] The lower and higher values of Cd are divided into two groups:

(i) The first group of samples that included SP_1, SP_3, SP_4, SP_5, and SP_6 contained high concentrations of Cd (6.05–10.40 mg kg^{-1}) and is presented in category B of sediment pollution (Table 5).

(ii) The second group of samples that included SP_2, SP_7, and SP_8 contained low concentrations of Cd (3.73–4.31 mg kg^{-1}), which is presented in category A of sediment unpolluted (Table 5).

In Përlepnica Lake, the average concentration of Cd was 6.32 mg kg^{-1}; Badovc Lake (Kosovo), Balaton Lake (Hungary), and Skadar Lake (Albania) concentrations of Cd were low (Table 6).

Copper (Cu) causes toxic effects such as liver damage, Wilson disease, insomnia, and gastrointestinal problems.[32] The lower and higher values of Cu are divided into two groups:

(i) The first group of samples that included SP_1, SP_2, SP_3, SP_4, SP_5, SP_6, and SP_8 contained high concentrations of Cu (52.31–89.99 mg kg^{-1}), and is presented in category B of sediment pollution (Table 5).

(ii) The second group of samples that included SP_7 contained a low concentration of Cu 40.24 mg kg^{-1}, and is presented in category A of sediment unpolluted (Table 5).

The average concentration of Cu in Përlepnica Lake sediment was 61.08 mg kg^{-1}. It was almost the same value as in Badovc Lake where the average concentration of Cu was 61.21 mg kg^{-1}, and the concentration of these two lakes was much higher compared to Balaton and Skadar Lake (Table 6).

Manganese (Mn) is considered an essential micronutrient for the metabolic process. Its toxicity alters physiological, biochemical, and molecular processes at the cell level.[33,34] The values of Mn in all samples were very high (505.56–930.92 mg kg^{-1}) and the samples were included in a single group in category C of sediment significant pollution (Table 5).

Nevertheless, Mn according to "The Dutch List" has no criterion value because it is a massive element in soil, but the average concentration of Mn (708.65 mg kg^{-1}) in Përlepnica Lake was very high compared to the Badovc Lake Mn (660 mg kg^{-1}), while in Balaton Lake (Hungary) and Skadar Lake the concentration of Mn in the sediments was not present (Table 6).

Nickel (Ni) has toxic effects causing dermatitis, nausea, chronic asthma, coughing, a human carcinogen, reproductive effects, and respiratory cancer.[32] Concentrations of Ni in all samples were low (31.46–44.62 mg kg^{-1}) and the samples were included in a single group in category A of sediment unpolluted (Table 5).

In Përlepnica Lake, the average concentration of Ni in sediment was 37.27 mg kg^{-1}, while in Badovc Lake the aver-

Table 6 – Comparison of metal concentration in sediments of Përlepnica Lake, Badovc Lake, Balaton Lake, and Skadar Lake
Tablica 6 – Usporedba koncentracije metala u sedimentu Përlepničkog jezera te jezera Badovac, jezera Balaton i Skadarskog jezera

Metals	Unit	Përlepnica Lake (Kosovo), results of this study	Badovc Lake (Kosovo) Results/ average[37]	Lake Balaton (Hungary) min-max[37]	Lake Skadar (Albania) min-max[37]
Fe	$mg\,kg^{-1}$	1.83	19084	–	–
Cr		71.43	276	5.70–66	10.28–82.60
Cu		61.08	61.20	0.70–36	7.65–28.53
Cd		6.32	–	0.10–0.70	0.05–1.01
Mn		708.65	660	–	–
Ni		37.27	305	4.40–5.50	23.60–136.10
Zn		604.55	122	13–150	25.70–87.20
Pb		264.26	167	2.40–160	3.71–60.10

age concentration of Ni was 305 $mg\,kg^{-1}$, and this concentration was very high compared to Përlepnica, Balaton, and Skadar lakes (Table 6).

Lead (Pb) is a highly toxic pollutant, and its effects on the human body are devastating.[35] The values of Pb in all samples were very high (123.38–346.60 $mg\,kg^{-1}$) and the samples were included in a single group as presented in category B of sediment significant pollution (Table 5).

The average concentration of Pb in the sediment of Përlepnica Lake was 264.26 $mg\,kg^{-1}$, a very high concentration compared to Badovc, Balaton, and Skadar lakes (Table 6).

Zinc (Zn) has toxic effects on the nervous system and its fumes can cause skin problems.[36] Values of Zn in all samples were very high (300.68–927.76 $mg\,kg^{-1}$) and the samples were included in a single group as presented in category C of sediment significant pollution (Table 5).

The concentration of Zn in Përlepnica Lake was quite high, and the average concentration in sediments was 604.55 $mg\,kg^{-1}$, while in Badovc Lake the average concentration of Zn was 122 $mg\,kg^{-1}$. Balaton and Skadar lakes Zn concentration was not high (Table 6).

Mercury (Hg) is recognized as a toxic, persistent, and mobile contaminant that does not degrade into the environment.[36] Concentration of Hg in the sediment in samples SP_1–SP_8 was detected at the level of 1 ppb, and fortunately did not appear in the sediment of Përlepnica Lake (Table 5).

According to this study, it can be assumed that the sediment of Përlepnica Lake has higher sediment mass compared to the other lakes mentioned.

4.3 Metal concentration in water

Sediments have one of the most important roles in the water environment, as they are usually the main collector

Table 7 – Concentration of heavy metals in the water of Përlepnica Lake (March, April, and June 2020)
Tablica 7 – Koncentracija teških metala u vodi jezera Përlepnica (ožujak, travanj i lipanj 2020.)

Samples	Unit	Metals							
		Mn	Fe	Zn	Cu	Cr	Pb	Cd	Ni
SP_1	$mg\,l^{-1}$	0.003	0.31	0.09	0.022	0.038	*bdl	0.002	*bdl
SP_2		0.002	0.31	0.08	0.021	0.036	*bdl	0.002	*bdl
SP_3		0.003	0.27	0.05	0.018	0.035	*bdl	0.004	*bdl
SP_4		0.003	0.27	0.05	0.017	0.035	*bdl	0.004	*bdl
SP_5		0.004	0.29	0.06	0.021	0.034	*bdl	0.004	*bdl
SP_6		0.004	0.30	0.05	0.021	0.033	*bdl	0.003	*bdl
SP_1–SP_6 Average (June 2020)		0.003	0.295	0.066	0.020	0.035	*bdl	0.003	*bdl
SP_1–SP_6 average (March 2020)		0.003	0.310	0.07	0.019	0.039	*bdl	0.003	*bdl
SP_1–SP_6 average (April 2020)		0.002	0.295	0.066	0.018	0.035	*bdl	0.003	*bdl
EU 75/440		0.05	0.10	0.50	0.02	0.05	0.01	0.005	0.02
Standard Deviation (SD)**		0.0026	0.02	0.016	0.002	0.0026	–	0.001	–

* Below detection level

** SD Average (June-March-April) 2020

of contaminants and pollutants introduced into water systems – substances dissolved in water are bonded on the solid particles over time, and finally trapped in bottom deposits.[38] The amount of available ecological water is a key factor that determines the function of the ecosystem.[39] Also, the monitoring of heavy metals is extremely important in an aquatic environment used by the population for drinking water. The concentrations of HMs in this lake with the analysed samples SP_1–SP_6 were in accordance with EC 75/440: Mn 0.002–0.004 mg l^{-1}, Zn 0.051–0.091 mg l^{-1}, Cr 0.033–0.038 mg l^{-1}, Cd 0.002–0.004 mg l^{-1}, Pb < 1 ppb, and Ni < 1 ppb (Table 7).

Concentration of Fe (0.271–0.316 mg l^{-1}) in samples SP_1–SP_6 was not in accordance with EC 75/440 (Table 7). The concentration of Cu in some samples was above the concentration permitted by Directive EC 75/440. Thus, they were divided into two groups:

(i) samples SP_3 and SP_4 were in accordance with EC Directive 75/440 (Table 7).

(ii) samples SP_1, SP_2, SP_5, and SP_6 were not in accordance with EC directive 75/440 (Table 7)

From the analysed water and sediment samples, the concentration of heavy metals in the sediments is beginning to penetrate the water of Përlepnica Lake. This will affect the consumers of this water. Table 8 shows the comparison of HMs concentrations in the water of Përlepnica, Badovc, and Batlava Lakes.

Table 8 – Comparison of metal concentrations in waters of Përlepnica, Badovc, and Batlava lakes
Tablica 8 – Usporedba koncentracije metala u vodama jezera Përlepnica, Badovac i Batlava

Metals	Unit	Përlepnica Lake (Kosovo), current study – results/average	Badovc Lake (Kosovo) – results/average[37]	Batlava Lake (Kosovo) – results/average[37]
Fe	mg l^{-1}	0.29	0.99	0.23
Cr		0.035	<0.001	0.0016
Cu		0.020	0.031	0.001
Cd		0.003	<0.001	–
Mn		0.003	0.096	0.024
Ni		–	<0.01	<0.01
Zn		0.066	0.002	0.053
Pb		–	<0.01	<0.01

5 Conclusions

In the current study, several HMs (Fe, Cd, Cr, Cu, Mn, Ni, Pb, Zn, and Hg) were determined to cause future problems for the aquatic environment and the inhabitants of this municipality. In the water, the average concentration of Fe was 0.29–0.31 mg l^{-1}, Cu was above 0.02 mg l^{-1}, and Zn was 0.06–0.07 mg l^{-1}. The water analyses of Përlepnica Lake showed that the concentrations of Fe, Cu, and Zn in the water are not in the accordance with EU Directive 75/440.

In addition, sediment analysis showed that the average content of the HMs was as follows: Pb – 264.26 mg kg^{-1}, Fe – 1.83 mg kg^{-1}, Cd – 6.32 mg kg^{-1}, Cu – 61.08 mg kg^{-1}, Mn – 708.65 mg kg^{-1}, and Zn – 604.55 mg kg^{-1}, which exceeded the permissible limits according to the instructions of the New Dutch List. From the results obtained in sediment and water, it is preferable to continuously monitor the lake, because many HMs are too close to penetrate the water and thus start water pollution of Përlepnica Lake. In addition, according to the results obtained in the analytical laboratory for heavy metals in water and sediments, the authors propose that the bed (bottom) of Përlepnica Lake should be cleaned or the sediment mechanically removed, as it contains many heavy metals and can cause serious problems soon.

List of abbreviations
Popis kratica

HMs – heavy metals
– teški metali

ICP-OES – inductively coupled plasma-optical emission spectrometry
– induktivno spregnuta plazma-optička emisijska spektrometrija

AAS – atomic absorption spectrometry
– atomska apsorpcijska spektrometrija

EU – European Union
– Europska unija

ISO – International Organization for Standardization
– Međunarodna organizacija za standardizaciju

EDTA – ethylenediaminetetraacetic acid
– etilendiamintetraoctena kiselina

CW – conductivity of water
– vodljivost vode

GH – general hardness
– ukupna tvrdoća

CS – carbonic strength
– ugljična čvrstoća

NTU – nephelometric turbidity unit
– nefelometrijska jedinica zamućenja

SD – standard deviation
– standardna devijacija

SP – sample
– uzorak

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SAŽETAK

Koncentracija teških metala u sedimentu i vodi jezera Përlepnica, Kosovo

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Gjilan je grad na jugoistoku Kosova kojem glavnu opskrbu pitkom vodom čini jezero Përlepnica. U jezeru su analizirani teški metali u sedimentu i vodi te fizikalno-kemijski parametri. Utvrđene prosječne koncentracije metala u sedimentu slijedile su niz: Mn (708,65 mg kg^{-1}) > Zn (604,55 mg kg^{-1}) > Pb (264,26 mg kg^{-1}) > Cr (71,43 mg kg^{-1}) > Cu (61,08 mg kg^{-1}) > Ni (37,27 mg kg^{-1}) > Cd (6,32 mg kg^{-1}) > Fe (1,83 mg kg^{-1}). Prosječne koncentracije metala u vodi slijedile su niz: Fe (0,295 mg l^{-1}) > Zn (0,066 mg l^{-1}) > Cu (0,020 mg l^{-1}) > Cr (0,035 mg l^{-1}) > Cd (0,003 mg l^{-1}) > Mn (0,003 mg l^{-1}). Koncentracije teških metala u sedimentu, kao što su Cd, Cu, Mn, Pb i Zn, nisu bile u skladu s kriterijima koji se primjenjuju za kontaminirana područja u Nizozemskoj, a koncentracije Fe i Cu u vodi nisu bile u skladu s direktivom EU 75/440.

Ključne riječi
Teški metali, jezero Përlepnica, voda, sediment, onečišćenje, ICP-OES, AAS

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