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Temporal Variation of Metals in Water, Sediment and Tissues of the European Chup (*Squalius cephalus* L.)

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Abstract The aim of this study was to analyze seasonal variation of levels of Chromium (Cr), Manganese (Mn), Nickel (Ni), Copper (Cu), Zinc (Zn), Cadmium (Cd) and Lead (Pb) in water, sediment and tissues of *Squalius cephalus* (L., 1758) taken from five different stations chosen at Yamula Dam Lake (Kayseri, Turkey). Concentrations of metals were generally sorted as water<tissue<sediment. The highest concentration of metals in water was observed in the samples taken in the summer (Mn, 1.04 ± 0.15 mg L^{-1}; Ni, 0.42 ± 0.12 mg L^{-1}; Cu, 1.25 ± 0.88 mg L^{-1}; Zn, 3.61 ± 1.53 mg L^{-1}; Pb, 0.58 ± 0.09 mg L^{-1}). While the highest Zn (24.85 ± 21.82 µg g^{-1}) and Pb (10.89 ± 4.2 µg g^{-1}) concentrations in sediment were observed in the winter samples, the highest Mn concentration (167.2 ± 99.37 µg g^{-1}) was observed in the summer. Cd pollution was determined in sediment according to international criteria. The highest metal concentration in tissues was generally observed in the liver and the other tissues; following liver were gills and muscles. In liver tissues, while the highest accumulation of Zn (110.34 ± 13.1) and Mn (22.5 ± 14.85 µg g^{-1}), which are essential for the body, were observed in the winter, Pb (22.58 ± 7.83 µg g^{-1}) and Cd (11.77 ± 7.83 µg g^{-1}), which are toxic, were found to be higher in the summer. Also, concentrations of Mn, Cd and Pb in muscle tissues were found to be above the limits permitted.

Keywords Metal · Seasonal variation · Water sediment · *Squalius cephalus*

Overproduction due to an increase in human population brings with it an increase in the amount of waste left to nature. When the ratio of this increase exceeds the self-renewal capacity of nature, the environment becomes polluted. The polluters found in soil and air eventually reaches the water, causing water pollution. In recent years, industrial wastes and sewage carrying urban wastes pollute rivers and lakes into which they are discharged as a result of the development of technology, and this threatens living organisms in aqueous environments. In terms of the use of drain water for irrigation and the organisms living in the environment for drain water discharge, especially trace elements in drain water are important in terms of public health, because trace elements are directly and indirectly transferred via the food chain. More importantly, the combination of toxic organic wastes with metals or the changing of them into other compounds can cause problems due to the formation of more toxic wastes (Ural et al. 2012). Although heavy metals are found at low levels in water, they can reach significant levels in sediment and aqueous biota. Also, sediment can be the end point of trace elements in freshwater ecosystems and it can serve as an archive in the studies of metals. However, all forms of heavy metals are not available. Also, the presence and form of heavy metal in sediment can differ according to pH, electrical conductivity, total organic material amount, and redox potential (Karadede and Unlu 2000). Starting from primary producers, heavy metals reach the fish via other consumer organisms in water. Fish are important sources of proteins and polyunsaturated fatty acids in human nutrition. The determination of metal accumulation in fish in parallel to heavy metal pollution is an important

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issue in terms of both fish biology and human health. Therefore, many studies have been carried out on the accumulation of heavy metals in fish and other organisms in the aqueous environment (Aktumsek and Gezgin 2011; Karadede and Unlu 2000; Zyadah and Abdel-Baky 2000). The concentration of heavy metal in fish is related to the nutritional habit of the fish species, age, environment etc. and it can differ between tissues and organs. In this study, we aimed to determine seasonal variation of heavy metal levels in water, sediment and fish tissue samples statistically and if any difference was observed, to find the reasons for these.

Materials and Methods

The study was carried out at 5 different stations chosen at the Yamula Dam Lake (Kayseri, Turkey) in the period between April 2009 and January 2010 (Fig. 1). The samples were taken from the stations in July, October, January and April, which were months chosen to represent the seasons. Before the study, all materials used were washed with the solution prepared with nitric acid and deionized water and then they were dried in a drying-oven to prevent any contamination. The glass bottles had been kept in 10 % HNO_3 overnight and washed twice with the dam water in the sampling field. Water samples from each station were taken at 0.5 m depth and placed in 1 L glass bottles. Then, the water samples were filtered (45 μm Whatman no. 1 filter paper) in the lab and 9 mL of the water was kept in a falcon tube and stored in a refrigerator until analysis, after the addition of 1 mL HNO_3.

Sediment from each station was taken at 1.5 m distance from the lakeshore and a depth of 15 cm from the surface.

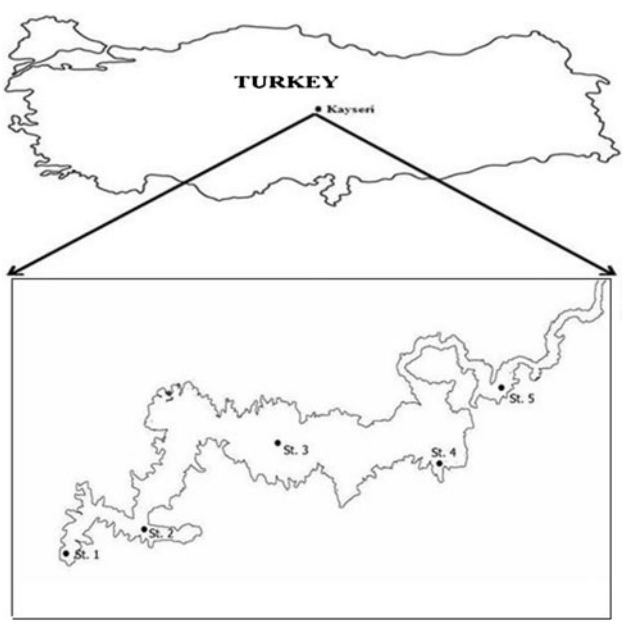

Fig. 1 Map of study area (St: station)

The sediment samples were dried by mixing at intervals at room temperature for 7 days and sieved through 2 mm sieve. Dried samples were properly crushed into powder in a porcelain mortar. Samples were digested in a microwave digestion system (CEM Mars 5; CEM Corporation Mathews, NC, USA) (Karadede and Unlu 2000). Fish samples were provided by local fisherman and brought to the lab in ice buckets on the same day. For fish samples, the eatable muscle tissue, liver tissue and gills were separated as 4 g each and dried at 80°C for 3 days. The samples were digested in a microwave oven and kept at +4°C until analysis.

The total concentration of metals was determined using an inductively coupled plasma mass spectrometer (Agilent, 7500a). Standard readings were performed after every 10 samples to determine the stability of the device. A blind solution was prepared to determine the analytical method and possible contamination. The detection limits of Cr, Mn, Ni, Cu, Zn, Cd and Pb were 0.07, 0.1, 0.17, 0.82, 0.84, 0.04 and 0.16 ng mL^{-1}, respectively. The samples were analyzed in triplicate. All chemicals used in this study were analytical reagent grade (Merck. Darmstadt. Germany). The values showed the mean with the standard deviations of triplicate-sample analysis. Kolmogrov–Simirnov and Levene's tests were applied to control the normal distribution and homogeneity of the values. When heterogeneity was present, logarithmic transformation ($\ln(x + 1)$) was applied and homogeneity was again determined. One-way analysis of variance was applied to test the significance of the difference between seasons and tissues. All statistical analyses were carried out by SPSS 15.00 packaged software. Significance degree was accepted as $p < 0.05$.

Results and Discussion

The seasonal mean of metal concentrations in water and sediments samples taken from Yamula Dam are given in Tables 1 and 2. No significant difference was seasonally observed for Cr and Cd, as seen in Table 1. The highest metal concentrations of Mn, Ni, Zn and Pb were observed in the summer. It was found that the concentration of Cu was higher in the summer and autumn than in the spring and winter.

Statistically significant difference was not observed in terms of Cr, Ni and Cu concentrations in sediment samples (Table 2). While the highest Mn concentration was observed in the summer, the concentrations of Pb and Zn were the highest in the winter. In contrast, the Cd concentration was the lowest in the summer and a statistically significant difference among metal concentrations in the other seasons was not found.

The seasonal variation of metal accumulation in fish tissues is given in Table 3. According to Table 3, no

Table 1 Seasonal mean (±SD) concentrations of element in water (mg L^{-1})

	Cr	Mn	Ni	Cu	Zn	Cd	Pb
Spring	0.27 ± 0.01^{A}	0.18 ± 0.08^{A}	0.25 ± 0.16^{A}	0.25 ± 0.02^{A}	0.67 ± 0.08^{A}	0.16 ± 0.009^{A}	0.28 ± 0.01^{A}
Summer	0.27 ± 0.02^{A}	1.04 ± 0.15^{B}	0.42 ± 0.12^{B}	1.25 ± 0.88^{B}	3.61 ± 1.53^{B}	0.17 ± 0.01^{A}	0.58 ± 0.09^{C}
Autumn	0.27 ± 0.01^{A}	0.14 ± 0.01^{A}	0.28 ± 0.18^{A}	0.85 ± 0.26^{B}	1.26 ± 0.68^{A}	0.16 ± 0.009^{A}	0.35 ± 0.02^{B}
Winter	0.27 ± 0.01^{A}	0.29 ± 0.2^{A}	0.19 ± 0.01^{A}	0.39 ± 0.04^{A}	0.86 ± 0.15^{A}	0.16 ± 0.008^{A}	0.36 ± 0.04^{B}

* Different letters in same column indicate significant differences at $p < 0.05$ (ANOVA). *SD* standard deviation

Table 2 Seasonal mean (± SD) concentrations of element in sediment (µg g^{-1})

	Cr	Mn	Ni	Cu	Zn	Cd	Pb
Spring	7.16 ± 2.74^{A}	90.7 ± 24.72^{A}	12 ± 6.55^{A}	7.32 ± 0.771^{A}	11.02 ± 4.97^{A}	4.43 ± 0.26^{B}	8.26 ± 1.44^{A}
Summer	10.05 ± 5.89^{A}	167.2 ± 99.37^{B}	19.02 ± 13.68^{A}	7.35 ± 2.848^{A}	19.43 ± 11.96^{AB}	4.03 ± 0.3^{A}	6.00 ± 1.16^{A}
Autumn	10.93 ± 4.25^{A}	128.2 ± 63.04^{AB}	20.07 ± 8.94^{A}	7.88 ± 2.734^{A}	12.52 ± 2.88^{AB}	4.69 ± 0.23^{B}	7.18 ± 1.03^{A}
Winter	7.66 ± 1.38^{A}	96.9 ± 26.26^{A}	12.84 ± 2.11^{A}	8.19 ± 1.716^{A}	24.85 ± 21.82^{B}	4.51 ± 0.3^{B}	10.89 ± 4.2^{B}

* Different letters in same column indicate significant differences at $p < 0.05$ (ANOVA). *SD* standard deviation

statistically significant difference was seasonally observed in terms of Ni, Zn and Pb metals in muscle tissues. While Cr, Mn and Cu concentrations were high in the winter in muscle tissues, Cd was lower in the winter than in the other seasons. While Cr, Mn and Zn showed the highest accumulation in the winter in liver tissues, Cd and Pb showed the highest accumulation in the summer. There was no difference in accumulated Cu concentration in the liver in terms of seasons. While the lowest Ni accumulation was observed in the spring, a statistically significant difference with the other seasons was not found. While Cr, Mn and Cu in gills showed statistically the highest accumulation in the winter, the lowest Zn accumulation was observed in the spring and a statistically significant difference among the other seasons was not found (Table 3). In contrast, the highest Pb accumulation was observed in the winter. No statistically significant difference was found among Cr, Mn, Ni, Zn and Cd concentrations when metal concentrations in tissues were compared according to the spring (Table 4). While the highest Cu accumulation was in liver in the summer, Pb was accumulated in gills. It was found that while the highest accumulations of Cr, Ni, Cu and Cd were observed in the liver in the summer, the lowest Pb accumulation was found in the gills and the lowest Zn accumulation was observed in muscle tissues. Mn mostly accumulated in gills. In the autumn, while Cr, Ni, Cu, Cd and Pb mostly accumulated in the liver, over accumulation occurred in the liver tissues for all metals. The lowest accumulations of Zn and Mn were determined in muscle tissues. In the winter, Cr, Ni, Zn and Cd had the highest accumulation in the liver. While no difference for Mn was found among tissues, the lowest Cu accumulation was observed in muscle tissues and the lowest Pb accumulation was observed in the gills.

In this study, annual mean of metal concentrations in water was determined for Cr, Mn, Ni, Zn, Cu, Cd and Pb as 0.27, 0.41, 0.28, 0.68, 1.6, 0.16, 0.39 mgL^{-1}, respectively. As seen in Table 5, Cr and Ni values in Abant and Sapanca Lakes were found to be higher than the values in the present study, but metal concentrations determined in other studies were found lower than our findings. Similarly, all metal values were found higher than references according to the criteria published by international organizations.

The reason for this can be an increase in evaporation in the summer and increase in metal concentrations due to a decrease in water amount. Similar findings had found in a study which conducted Beysehir Dam Lake (Tekin-Ozan and Kir 2008). Yamula Dam is the first dam lake set up on the river and this can be the reason for the metal concentrations being higher in Yamula dam water samples than in other studies. Also, the sites of the stations in the study that are near to places having intense agricultural activities and unprocessed domestic wastes flowing into the dam can have effects on the concentrations. In this study, all metal concentrations except Cr and Cd were statistically higher in the summer than in the other seasons.

According to Table 5, Cr concentration in sediment was higher than in Demirkopru Dam and but it was lower than in Texoma Lake. While Ni concentration was only higher than in Demirkopru Dam, it was found to be lower than in other studies. The concentration of Zn was found to be lower than in all other studies. Pb and Cd concentrations were found to be higher than in the other lakes. In a similar study carried out by Landajo et al. (2004) they found that metal concentrations were lower in sediment taken in the winter than in the summer. However, in the study carried out in Tigris River Karadede-Akin and Unlu (2007) found that all metal concentrations in sediment samples were high

Table 3 Statistical comparation of mean tissue concentrations (μg g^{-1}dry weight) of metals according to different seasons together with standard deviation (SD)

	Cr	Mn	Ni	Cu	Zn	Cd	Pb
M							
Sp	$2.5 \pm 0.4^{A*}$	7.8 ± 2.4^{A}	3.3 ± 0.4^{A}	4.9 ± 0.7^{A}	28.9 ± 15.6^{A}	4.7 ± 0.6^{B}	9.1 ± 0.8^{A}
Su	2.5 ± 0.4^{A}	6.5 ± 3.7^{A}	3.2 ± 0.6^{A}	4.7 ± 0.8^{A}	22.9 ± 20.00^{A}	4.7 ± 0.4^{B}	9.4 ± 3.1^{A}
Au	2.5 ± 0.2^{A}	5.6 ± 3.5^{A}	3.3 ± 0.5^{A}	3.7 ± 0.9^{A}	31.4 ± 17.2^{A}	4.5 ± 0.3^{B}	9.4 ± 6.3^{A}
Wi	3.6 ± 1.3^{B}	13.6 ± 8.5^{B}	3.1 ± 0.4^{A}	7.8 ± 5.2^{B}	28.1 ± 10.5^{A}	3.7 ± 0.6^{A}	7.6 ± 2.3^{A}
L							
Sp	2.9 ± 1.2^{A}	6.1 ± 2.3^{A}	3.9 ± 1.5^{A}	20.1 ± 18.2^{A}	30.4 ± 10.5^{A}	6.0 ± 2.9^{A}	9.1 ± 0.8^{A}
Su	5.7 ± 3.6^{AB}	12.4 ± 6.7^{A}	8.7 ± 4.3^{B}	9.5 ± 6.9^{A}	69.1 ± 43.7^{B}	11.8 ± 7.8^{B}	22.6 ± 7.8^{B}
Au	5.0 ± 1.8^{AB}	15.0 ± 5.3^{AB}	7.6 ± 2.9^{B}	8.8 ± 2.3^{A}	72.4 ± 34.5^{B}	8.5 ± 2.7^{AB}	14.1 ± 2.7^{AB}
Wi	8.0 ± 2.3^{BC}	22.5 ± 14.8^{B}	7.5 ± 1.7^{B}	17.8 ± 10.7^{A}	110.3 ± 13.1^{C}	7.9 ± 3.8^{AB}	15.8 ± 3.8^{AB}
G							
Sp	2.4 ± 0.2^{A}	7.1 ± 3.1^{A}	3.3 ± 0.4^{A}	4.0 ± 1.3^{A}	21.2 ± 10.9^{A}	4.9 ± 0.3^{B}	12.9 ± 11.6^{B}
Su	2.9 ± 0.8^{A}	13.5 ± 7.8^{A}	3.2 ± 0.6^{A}	4.4 ± 1.6^{A}	56.5 ± 17.3^{B}	5.0 ± 0.5^{B}	5.1 ± 0.5^{A}
Au	3.1 ± 0.7^{A}	15.0 ± 9.8^{A}	4.2 ± 1.2^{B}	3.7 ± 0.9^{A}	53.7 ± 20.7^{B}	4.6 ± 0.4^{B}	4.6 ± 0.4^{A}
Wi	4.0 ± 1.3^{B}	25.6 ± 13.8^{B}	3.8 ± 0.3^{AB}	12.2 ± 6.6^{B}	46.2 ± 16.4^{B}	4.0 ± 0.7^{A}	4.1 ± 0.7^{A}

* Different letters in each group indicate significant differences at $p < 0.05$ (ANOVA). *M* muscle, *L* liver, *G* gill

Table 4 Seasonal mean concentrations (μg g^{-1}dry weight) of metals in tissue of *S. cephalus* together with standard deviation (SD)

	Cr	Mn	Ni	Cu	Zn	Cd	Pb
Spring							
M	2.5 ± 0.4^{a}	7.8 ± 2.4^{a}	3.3 ± 0.4^{a}	4.9 ± 0.7^{a}	28.9 ± 15.6^{a}	4.7 ± 0.6^{a}	9.1 ± 0.8^{a}
L	2.9 ± 1.2^{a}	6.1 ± 2.3^{a}	3.9 ± 1.5^{a}	20.1 ± 18.2^{b}	30.4 ± 10.5^{a}	6.0 ± 2.9^{a}	9.2 ± 2.9^{a}
G	2.4 ± 0.2^{a}	7.1 ± 3.1^{a}	3.3 ± 0.4^{a}	4.0 ± 1.3^{a}	21.2 ± 10.9^{a}	4.9 ± 0.3^{a}	12.9 ± 11.6^{b}
Summer							
M	2.5 ± 0.4^{a}	6.5 ± 3.7^{a}	3.2 ± 0.6^{a}	4.7 ± 0.8^{a}	22.9 ± 20.0^{a}	4.7 ± 0.4^{a}	9.4 ± 3.1^{b}
L	5.7 ± 3.6^{b}	12.4 ± 6.7^{ab}	8.7 ± 4.3^{b}	9.5 ± 6.9^{b}	69.1 ± 43.7^{b}	11.8 ± 7.8^{b}	22.6 ± 7.8^{b}
G	2.9 ± 0.8^{a}	13.5 ± 7.8^{b}	3.2 ± 0.6^{a}	4.4 ± 1.6^{a}	56.5 ± 17.3^{b}	5.0 ± 0.5^{a}	5.1 ± 0.5^{a}
Autumn							
M	2.5 ± 0.2^{a}	5.6 ± 3.5^{a}	3.3 ± 0.5^{a}	3.7 ± 0.9^{a}	31.4 ± 17.2^{a}	4.5 ± 0.2^{a}	9.4 ± 6.3^{b}
L	5.0 ± 1.8^{b}	15.0 ± 5.3^{b}	7.6 ± 2.9^{b}	8.8 ± 2.3^{b}	72.4 ± 34.5^{b}	8.5 ± 2.7^{b}	14.1 ± 2.7^{b}
G	3.1 ± 0.7^{a}	15.0 ± 9.8^{b}	4.2 ± 1.2^{a}	3.7 ± 0.9^{a}	53.7 ± 20.7^{ab}	4.6 ± 0.4^{a}	4.6 ± 0.4^{a}
Winter							
M	3.6 ± 1.3^{a}	13.6 ± 8.5^{a}	3.1 ± 0.4^{a}	7.8 ± 5.2^{a}	28.1 ± 10.5^{a}	3.6 ± 0.6^{a}	7.6 ± 2.3^{b}
L	8.0 ± 2.3^{b}	22.5 ± 14.8^{a}	7.5 ± 1.7^{b}	17.8 ± 10.7^{b}	110.3 ± 13.1^{c}	7.9 ± 3.8^{b}	15.8 ± 3.8^{b}
G	4.0 ± 1.3^{a}	25.6 ± 13.8^{a}	3.8 ± 0.3^{a}	12.2 ± 6.6^{ab}	46.2 ± 16.4^{b}	4.0 ± 0.7^{a}	4.1 ± 0.7^{a}

* Different letters in each group indicate significant differences at $p < 0.05$ (ANOVA). *M* muscle, *L* liver, *G* gill)

in the summer. In the present study, no statistically significant difference was found among the seasons in terms of Cr, Ni and Cu concentrations. It was seen that the highest Zn and Pb concentrations were observed in the winter and the lowest amount was observed in the summer. In contrast, it was found that Mn and Cd concentrations were high in the summer and low in the winter. Yuzeroglu et al. (2010) suggested that the reason for the increase in metal accumulation in sediment after rainy seasons can be the increase in metal concentration due to metals in soil entering the dam with rainwater. Our findings support the opinion of Yuzeroglu et al. In a study carried out in Manzalla Lake, heavy metal concentrations in sediments were higher than in water and it was reported that although there was no significant difference among the stations in terms of studied metal concentrations, there were seasonally significant variations (Abdel-Baky and Zyadah 1998). According to the results of the study, metal concentrations in sediment were higher than the concentrations in water. Yilmaz et al. have suggested that most suspended particles

Table 5 The metal concentration in the Yamula Dam's water (μg L^{-1}) and sediment (μg g^{-1}) comparison with guidelines and different literature

	Cr	Ni	Cu	Cd	Pb	References
Water						
TSE	0.05	0.2	0.02	0.005	0.01	
WHO	0.05	–	0.02	0.01	0.05	
EPA	0.05	–	–	0.01	0.05	
Demirkopru Dam	0.006	0.016	0.02	0.001	0.02	Ozturk et al. (2008)
Sapanca Lake	0.6	0.4	0.2	0.02	0.3	Duman et al. (2007b)
Abant Lake	0.6	0.5	0.2	0.02	0.3	Duman et al. (2007a)
This study	0.27	0.28	1.6	0.16	0.39	–
Sediment						
Demirkopru Dam	6.75	14.3	15.1	0.82	6.5	Ozturk et al. (2008)
Avşar Dam	14.48	29.99	29.98	0.76	2.44	Ozturk et al. (2009)
Balaton Lake	5.7–66	4.5–55	0.7–36	0.1–0.7	2.4–160	Nguyen et al. (2005)
Texoma Lake	30	17	38	2	10	An and Kampbell (2003)
This study	8.95	15.98	16.95	17.66	8.08	–

in water have a high affinity for binding to metals, and metals discharged into the water are precipitated into sediment by the formation of complexes with suspended particles, which can cause metal accumulation in sediment (Yılmaz et al. 2007). Our findings also support this suggestion.

According to the EPA, the permitted limits of metal concentrations Cr, Mn, Ni, Cu, Zn, Cd and Pb in eatable fish muscle tissues are 4.1; 0.12; 27; 54; 410; 1.4 and 2.7 μg g^{-1}, respectively. In this study, the metal concentrations in muscle tissues for Cr, Mn, Ni, Cu, Zn, Cd and Pb were 3.06; 8.63; 3.22; 3.77; 27.83;4.38 and 8.88 μg g^{-1}, respectively. Metal levels in fish tissues were found to be higher than in water. The reason for this is that metals are precipitated and accumulated in depths and these metals are accumulated in tissues and organs due to intake from sediment or nutrition by fish. According to the result of this study, metal accumulation in the gills and liver was higher than in muscle tissues. This can generally be related to the accumulation of heavy metals in nonlethal concentrations in the metabolically active organs of fish (Tekin-Ozan and Kir 2008). The reasons for variation in metal accumulation due to metal types can be explained as the required amounts are different and they have different metabolic rates. Also, metallothionein enzyme synthesized in the liver and capable of binding to excess metal during an increase in metal concentration can be the reason for the accumulation of metal in the liver (Karadede-Akin and Unlu 2007). In the study carried out with two different fish species (*Labeo rohita* and *Ctenopharyngodon idella*) in Bhopal Lake by Neetu Malik et al. they observed that generally all metals were highly accumulated in the liver and gills and this situation varied among the species (Malik et al. 2010). It is known that Zn is an essential element and it has effects on the accumulation of toxic metals such as Cd and Pb. In our study, it was observed that Zn accumulated in higher concentrations than other metals. The most important reason for high metal concentrations in the gills can be explained by the direct contact of gill tissues with water. Also, metals can react with mucus secreted by the gills. Tekin-Ozan and Kir (2008) observed that accumulated metal concentrations in *Tinca tinca* caught in the Beysehir Lake were higher in hot seasons than in cold seasons. Our findings are compatible with this result.

Consequently, in this dam lake, metal concentrations in water, sediment and fish samples may be reach unacceptable levels according to national and international criteria. Besides, metal concentrations in water, sediment and fish tissues can seasonally vary due to metal types. Our study showed that metal accumulation was higher in the liver than in other organs. While Zn and Mn in the liver, which are essential for the body, showed the highest accumulation in the winter, toxic Pb and Cd were found in high concentrations in the summer. The findings of this study can be useful for biomonitoring studies.

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