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ARTICLE

The effects of seasonal heavy-metal pollution of Ladik Lake on pike fish (*Esox lucius*)

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ABSTRACT

Ladik Lake (Samsun, Turkey) is a natural landscape under the threat of pollution because of urban, agricultural and industrial activities. In this study, accumulation of heavy metals (Al, Ba, Cr, Cu, Fe, Mn, Pb and Zn) in the tissues (muscle, liver and gill) of sediment dwelling pike fish and in different substrates of Ladik Lake were investigated. Seasonal haematological, biochemical and histopathological parameters of the carp are also reported. In general, heavy-metal levels in water and sediment samples were found in the highest level in summer and the lowest in autumn. Histopathologic changes in the tissues of the fish (liver and gill) were at the minimum in winter while it was highest in summer. Blood biochemical parameters exhibited higher level in summer in comparison with other seasons. Al, Ba, Cr, Mn and Zn levels in the tissues of the fish were in the order gill>liver>muscle in all seasons. The levels of Cu, Fe and Pb were in the sequence liver>gill>muscle. Cr, Cu, Pb and Zn levels were determined to be in high level in the tissues of the fish with respect to the literature values. The heavy-metal levels in Ladik Lake and the fish health need to be regularly monitored for a sustainable environmental health.

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Biochemical parameters; *Esox lucius*; heavy metals; histopathology; sediment; water

Introduction

Metal pollution in aquatic ecosystems is important due to its bioaccumulation, biomagnifications and human exposure to these pollutants (Majnoni et al. 2013). Such pollution may result from the domestic, industrial and agricultural wastes (Gupta et al. 2009). As a result of flowing waste water to these aquatic environment Cd, Cr, Cu, Pb and Zn are the heavy metals that affect both the animal and human health (Asuquo et al. 2004). It is reported that this metal contamination in aquatic environments positively correlates with cancer, thereby increasing the risk of cancer in humans (Sirot et al. 2009; Zhang et al. 2011). Fish with high-quality proteins and amino acids are most likely to be affected

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by this contamination although they are at the top of the food chain in the aquatic ecosystem (Ersoy and Çelik 2010; Prusty et al. 2011). Heavy metals are usually found in low level in the water. However, if there is a metal pollution in the water, the metal accumulation in the fish increases through insects, crustaceans, crayfish and benthic worms that the constituents of the aquatic food chain the fish over time. For this reason, it is inevitable that human health is indirectly affected (Chevrier et al. 2009). Metal accumulation differs from the type of the fish and the accumulation part of the fish (Görür et al. 2012; Petrović et al. 2013). Since the presence of the toxic substances in the water affects the water quality parameters, the haematological, biochemical, antioxidant and histopathological structures of the fish are adversely affected (Kavitha et al. 2010). Thus, biological monitoring techniques, such as haematological and biochemical variables, have become real instruments for assessing water quality and pollution and the general health status of aquatic organisms (Pimpao et al. 2007; Olufayo 2009; Li et al. 2011). Since blood parameters are a short and inexpensive method of determining the presence, type and severity of possible stress/disease in any organism, reporting the changes in blood parameters of fish exposed to adverse environmental conditions (Li et al. 2010; Vasylkiv et al. 2010, 2011) gives valuable information about both the organism health and environmental pollutants (Kreutz et al. 2011; Saravanan et al. 2011).

Heavy metals penetrate to the fish mostly through gills and through feeding (Moiseenko et al. 2005; Kennedy and Fraser 2011) and cause structural damage in the secondary lamellae of the gills and impaired gill permeability. This is an indication of the transition to anaerobic metabolism that manifests as a decrease in ATP levels and an increase in lactate levels under the heavy-metal effect (Tort et al. 1987). It has been reported that fish tissues exposed to pollutants exhibit several histological abnormalities, including cytosolic vacuolization, single cell necrosis, fibrosis, apoptosis and tumour elevation, dense intrahepatic haemorrhage within a few hours and deterioration of the liver structure leading to death (Lezcano et al. 2012). Additionally, the metal accumulation in the fish muscle tissue, which is preferred for human consumption, induces histological changes, reduce glycogen content, changes in enzymatic activity, changes in carbohydrate metabolism, electrolyte levels and these result in reduced energetic resources, osmotic disturbances, disturbed neuro-muscular transmission and contraction, and finally, abnormal behaviour and body deformation (Jezierska and Witeska 2001).

In the aquatic environment the most important sources of heavy-metal accumulation in fish are benthic organisms and sediments. The accumulation of metal varies according to the type of the fish, feeding habits (carnivore), age, size, weight, sex and tissue (gills, liver, muscle) (De Wet et al. 1994; Carrasco et al. 2011; Squadrone et al. 2013; Has-Schön et al. 2015). Some metals such as copper (Cu), manganese (Mn), and zinc (Zn) are essential and play a significant role in biological systems. On the other hand, lead (Pb), cadmium (Cd) and mercury (Hg) are toxic elements in trace level. The essential metals may also be toxic at high concentrations as well. The most common contaminants in the aquatic environment are Cu, Pb, Zn, and Cd. Their levels in the water are commonly monitored (Wang et al. 2010).

The determination of the heavy-metal contents of the aquatic environments are usually examined by measuring heavy-metal concentrations in the water, sediment and various tissues (muscle, liver, gill, gonad, etc.) of aquatic organisms (Jezierska and Witeska 2001). Therefore, it is crucial to state that the purpose of this study is to investigate the seasonal change of the haematological, biochemical and histopathological parameters of the pike fish (*Esox lucius*), which has an economical significance, in Ladik Lake (Samsun, Turkey) and heavy metals in the water and the sediment.

Materials and methods

Research area

Ladik Lake (40°50′N to 41°00′N, 35°40′E to 36°05′E) is located within the borders of the Ladik district of Samsun Province in the central Black Sea region of Turkey (Bulut 2012). The lake is located on the 7th km of Erzincan highway, 10 km to the east of Ladik. Ladik Lake receives water from the streams Çakırgümüş and Kupecik coming from Akdağ and it empties its excess water into Tersakan Deresi being poured into Yeşilirmak River with one arm. The lake, which resembles an ellipse, has a length of 5 km, a width of 2 km, a depth of 2.5–6 m, an area of 10 km^2 and an altitude of 867 m (Anonim 2007; Uğurlu et al. 2009) (Figure 1). The lake is located within the tectonic Ladik collapse and is classified as eutrophic (Maraşlıoğlu 2001; Bulut 2012). The lake consists of animals and plants as well as floating islets, the rich peat mine and one of the most interesting natural sites (Bulut 2012).

Water analysis

Water samples were taken 1 L per two plastic bottles and then these were brought to the laboratory. Nitric acid was added at a rate of 5/1000 mL to the 1st plastic bottle to be

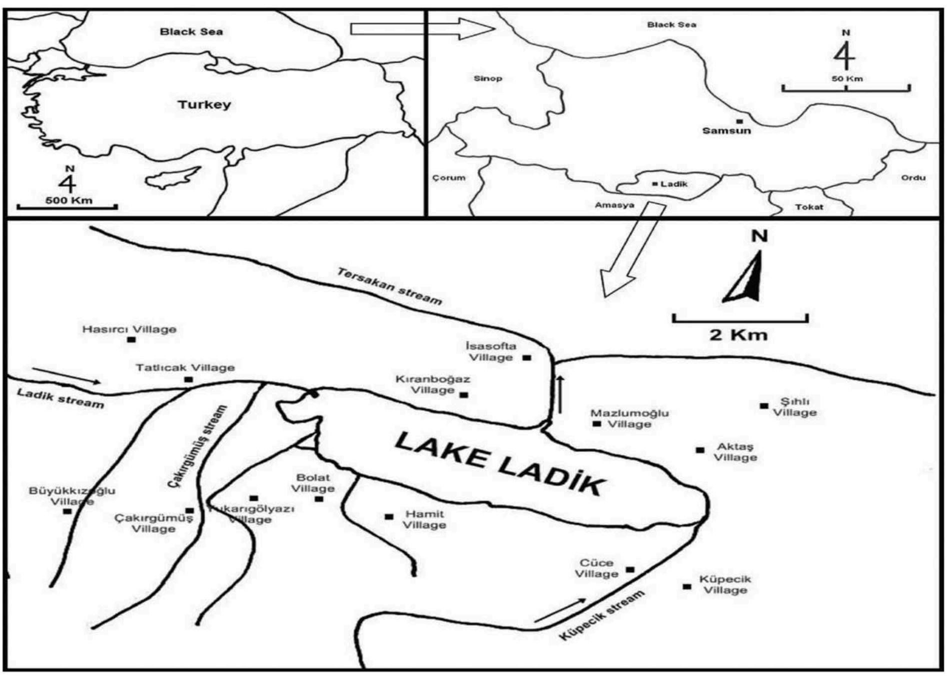

Figure 1. Map of Ladik Lake (Samsun, Turkey).

subjected to heavy-metal examination in the water. 40 mg/L $HgCl_2$ was added to the 2nd bottle to analyse the quality of the water by criteria (for keeping the nitrogen and phosphorus components stable). Analyses of the water samples were done twice with the indicated devices in accordance with the methods (TSI 2000, 2005; APHA 2005). The results are indicated in Table 1.

Sediment analysis

EPA 3051A Micronized acid digestion method was used in order to digest sediment with a dry weight of 0.5 g in a Milestone Start D brand microwave device by adding 9 mL HNO_3 (65%) and 3 mL HCl (37%). In the obtained samples, the analyses of the heavy metals (Al, Ba, Cr, Cu, Fe, Mn, Pb, Zn) were performed twice in Perkin Elmer Optima 2100 DV brand ICP-OES device by SM 3120 B ICP OES method (Subotić et al. 2013). The results were given as mean values.

Fish samples

The age, length, weight and sample counts of the pike fish that were caught with various sizes of fish net by the fishermen in the study is shown in Table 2. The age of pike was determined by scale reading (Epler et al. 2008; Žiliukienė and Žiliukas 2010; Yazıcıoğlu et al. 2016).

The field study was carried out in 2016. The water, sediment and fish samples were taken in January, April, July and October as well as the second month of each season from the Ladik Lake.

Tissue extraction

Fish tissues were processed by microwave digestion method. Fish tissue samples were digested with 2:1 ratio of nitric acid and perchloric acid, and then volumes made with double deionized water. Analysis of the heavy metals (Al, Ba, Cr, Cu, Fe, Mn, Pb, Zn) in fish tissues were performed by repeating twice according to SM 3120 B ICP-OES method (Perkinelmer Optima 2100 DV). The results were given as mean values.

Bioconcentration factor (BCF)

The bioconcentration factor is defined as the net result of the absorption, dissipation and removal of a substance from any organism after exposure through water and sediment. It was calculated as the ratio of the mean element level in the selected tissue (C_{fish}), expressed as μg g^{-1} wet weight, to the element concentration in water and sediment (C_{water} and $C_{sediment}$), expressed as mg L^{-1} and mg kg^{-1} BCF: $C_{fish\ tissue}/C_{water\ or\ sediment}$ (Subotić et al. 2013).

Serum biochemical analysis

At least 20 fish of each species were taken and blood was taken to the glass tubes from dorsal aorta by “direct tail cutting method”. Blood samples were centrifuged at +4°C and

Table 1. Methods used for chemical analyses in water, sediment and fish tissue.

Parameter	Method	Method number	Device brand/model
pH	Electrochemical method	SM 4500-H^+ B *	WTW/pH 330i/SET
Conductivity	Laboratory method	SM 2510 B *	WTW/pH 330i/SET
Total hardness	Account method	SM 2340 B *	–
Ammonium nitrogen (NH_4^+-N)	Titrimetric method	SM 4500- NH_3 B-C *	Velp Scientifica/UDK 127
Total organic carbon	Total organic carbon detection	TS 8195 EN 1484 **	Shimadzu/TOC-VCPN/TNM-1
Total nitrogen	Total nitrogen detection	TS EN 12260 ***	Shimadzu/TOC-VCPN/TNM-1
Anions (F^-, Cl^-, $NO2^-$, $NO3^-$, Br^-, PO_4^{3-}, SO_4^{2-})	Ion chromatography method	SM 4110 B *	Dionex/ICS 1000
Metals (Al, Ba, Cr, Cu, Fe, Mn, Pb, Zn)	ICP OES method	SM 3120 B *	Perkinelmer/Optima 2100 DV

* APHA (2005), Standard methods for the examination of water and wastewater, *American Public Health Association* Washington, DC, USA.
** TSI (2000), Water Quality – The guide for determination total organic carbon (TOC) and dissolved organic carbon (DOC), *Turkish Standards Institution*, Ankara, TURKEY.
*** TSI (2005), Water Quality – Nitrogen determination, Determination of bound nitrogen after oxidation to nitrogen dioxide, *Turkish Standards Institution*, Ankara, TURKEY.

Table 2. Minimum, maximum and mean length, weight and age of pike fish (*Esox lucius* Linnaeus, 1758) examined in the present study.

Seasons	Length (cm)	Weight (g)	Age (year)	Number of fish
Spring	57.1 ± 3.45 (53.7–60.5)	1565.5 ± 35.2 (1530–2105)	4.4 ± (4–5)	21
Summer	56.2 ± 3.66 (52.5–59.9)	1590.4 ± 42.6 (1570–1970)	4.3 ± (4–6)	24
Autumn	59.0 ± 4.11 (54.9–63.1)	1600.9 ± 61.7 (1490–1780)	4.5 ± (4–6)	20
Winter	58.7 ± 4.23 (54.5–62.9)	1570.6 ± 63.4 (1405–2050)	4.4 ± (4–5)	25

5000 rpm to obtain serum samples. Serum biochemical parameters [total protein, albumin, cholesterol (Col),triglyceride (Trg), blood urea nitrogen (BUN), glucose (Glu), alanine amino transferase (ALT), aspartate amino transferase (AST), lactate dehydrogenase (LDH), alkaline phosphatase (ALP), calcium (Ca), chloride (Cl), sodium (Na), potassium (K), iron (Fe), phosphorus (P)] were analysed with biochemical autoanalyzer (Siemens Adria 1800) using device-specific commercial kits within 12 h.

Analysis of haematological parameters

The blood was received from at least 20 fish from each group. The blood was taken to the EDTA tubes from the dorsal aorta by the "direct tail cutting method". Hematological parameters (total leukocyte, granulocyte and agranulocyte, erythrocyte, haemoglobin, haematocrit and thrombocyte) were analysed within 30 min by using automated veterinary haematology analyser MS 4e automated cell counter (Melet Schloesing Laboratories, France).

Histopathological examination of the tissues

Tissue specimens (liver and gill) taken from the fish species were placed in tissue tracking cassettes and then they were placed in freshly prepared fixative solution (10% formalin). The tissues were kept for 24 h fixation and then in tap water for another 24 h. Dehydration and paraffin impregnation processes were done by the Leica brand tissue monitoring device. Appropriate blocks were prepared by using a Leica brand tissue blocker from paraffin-impregnated tissues. Sections were taken from the prepared blocks with a thickness of 5 µm by Leica brand microtome. After getting the sections, the Harris Hematoxylin Eosin (HandE) staining was performed to show changes in the cellular structure. After the preparations were closed with entellan, they were examined on a Leica DM4000 light microscope.

Statistical analysis

Statistical analysis was carried out using the SPSS 15.0 statistical program (SPSS Inc., Chicago, IL, USA). All data were expressed as arithmetic means ± SD. For the analysis of the experimental parameters, one-way ANOVA followed by Duncan's multiple range test was used. The Pearson's correlation coefficient matrix for the elements was performed, as there was a linear relationship among the elements. Value of $p < 0.05$ was considered to be statistically significant.

Results

Distribution of water quality criteria according to seasons

The temperature was highest in the summer, and it was frozen parallel to the depth of the lake in the winter ($p < 0.05$). Depending on the temperature changes, the amount of dissolved oxygen varied and the highest values were recorded in winter months. Except the summer season, the pH of the lake water was 9.3 especially in the winter. The total organic carbon level was measured highest in the winter. Total nitrogen load was highest in the summer. Ammonium nitrogen was also found to be higher in the summer in comparison with other seasons. Phosphate content was at high level in the summer. Magnesium and calcium ions were at the highest level during the summer and spring seasons. Conductivity was highest in the summer and the lowest in the winter (Table 3).

Heavy-metal concentrations in water

All the heavy metals mentioned in the material and method were analysed but Al, Ba, Cr, Cu, Fe, Mn, Pb and Zn were determined after analysis. The seasonal distribution of heavy metals mentioned above in the Ladik Lake water samples were given in Table 4. According to this; the quantities of Al, Cr, Zn and Pb were determined as summer = spring>winter = autumn. Ba, Cu and Fe levels were determined as summer>spring = winter = autumn and Zn levels were determined as summer>spring>winter = autumn ($p < 0.05$).

Heavy-metal levels in sediment

The seasonal distributions of heavy metals (Al, Ba, Cr, Cu, Fe, Mn, Pb and Zn) in Ladik Lake sediment are presented in Table 5. According to this; Ba, Cu, Mn and Pb levels determined as summer = spring>winter = autumn, Fe levels determined as summer>spring = winter = autumn and Al levels determined as summer>spring>winter = autumn. There was no significant difference between Cr and Zn levels during the seasons ($p > 0.05$).

Table 3. Seasonal distribution of physico-chemical parameters of Ladik Lake (Samsun, Turkey) water.

	Seasons			
Parameters	Spring	Summer	Autumn	Winter
Temperature °C	17.4 ± 0.12^b	23.2 ± 0.40^a	15.4 ± 0.32^c	2.2 ± 0.21^d
Dissolved oxygen mg/L	7.6 ± 0.22^b	7.1 ± 0.15^c	8.2 ± 0.25^b	9.3 ± 0.20^a
pH	7.5 ± 0.03^b	7.2 ± 0.07^b	8.0 ± 0.08^a	8.1 ± 0.02^a
Total organic carbon (TOC) (mg/L)	21.9 ± 2.25^a	23.6 ± 1.47^a	15.1 ± 3.23^b	13.9 ± 1.11^b
Total organic nitrogen (mg/L) (TN)	1.5 ± 0.20^b	2.2 ± 0.21^a	1.4 ± 0.14^b	1.1 ± 0.17^c
Ammonium NH_4-N (mg/L)	0.4 ± 0.03^a	0.4 ± 0.03^a	0.3 ± 0.03^b	0.2 ± 0.02^c
Nitrite nitrogen NO_2-N (mg/L)	0.02 ± 0.003^b	0.04 ± 0.002^a	0.02 ± 0.005^b	0.02 ± 0.009^b
Nitrate nitrogen NO_3-N (mg/L)	3.9 ± 0.23^b	4.8 ± 0.07^a	1.8 ± 0.25^c	1.6 ± 0.59^c
PO_4-P (mg/L)	1.7 ± 0.16^a	1.5 ± 0.27^a	0.6 ± 0.05^b	0.4 ± 0.21^b
Fluoride (mg/L)	0.2 ± 0.001	0.3 ± 0.01	0.2 ± 0.01	0.2 ± 0.01
Magnesium Mg (mg/L)	5.1 ± 0.13^a	5.4 ± 0.12^a	2.3 ± 0.07^b	2.2 ± 0.09^b
Calcium Ca (mg/L)	36.4 ± 1.21^a	37.2 ± 2.41^a	27.7 ± 3.12^b	26.4 ± 2.08^b
Electrical conductivity (µS/cm)	295.0 ± 3.66^b	325.2 ± 2.35^a	277.8 ± 1.82^c	262.8 ± 2.75^d
Hardness (°dH)	110.2 ± 3.18^c	114.6 ± 3.25^b	104.4 ± 6.48^a	102.4 ± 3.51^b
Total dissolved solids (mg/L)	207.6 ± 4.13^b	168.6 ± 5.39^c	212.6 ± 1.12^b	241.0 ± 3.12^a

[a,b,c,d]The averages shown by the different letters on each line are statistically different ($p < 0.05$).
X ± SE: Mean ± standard error, number of samples sampled for each season: $N = 20$.

Table 4. Seasonal distribution of heavy-metal concentrations in water.

Metals (mg/L)	Seasons: Spring X± SE Min.–Max.	Summer X± SE Min–Max.	Autumn X± SE Min.–Max.	Winter X± SE Min.–Max.	The average annual
Al	0.5 ± 0.07[a] (0.4–0.6)	0.6 ± 0.03[a] (0.4–0.7)	0.1 ± 0.03[b] (0.1–0.2)	0.1 ± 0.03[b] (0.1–0.2)	0.32
Ba	0.04 ± 0.001[b] (0.03–0.04)	0.07 ± 0.006[a] (0.05–0.08)	0.03 ± 0.008[b] (0.02–0.04)	0.03 ± 0.007[b] (0.02–0.04)	0.04
Cr	0.04 ± 0.001[a] (0.03–0.06)	0.05 ± 0.002[a] (0.04–0.06)	0.02 ± 0.004[b] (0.01–0.03)	0.02 ± 0.004[b] (0.01–0.03)	0.03
Cu	0.5 ± 0.03[b] (0.3–0.8)	1.0 ± 0.04[a] (0.9–1.2)	0.4 ± 0.03[b] (0.2–0.6)	0.3 ± 0.04[b] (0.2–0.5)	0.55
Fe	11.3 ± 1.03[b] (6.6–11.7)	17.4 ± 1.17[a] (14.3–20.8)	7.32 ± 1.34[b] (6.2–9.4)	6.0 ± 1.22[b] (5.2–7.2)	10.50
Mn	0.9 ± 0.01[a] (0.5–1.6)	1.1 ± 0.001[a] (0.4–1.8)	0.2 ± 0.02[b] (0.1–0.3)	0.2 ± 0.01[b] (0.1–0.3)	0.6
Pb	0.4 ± 0.02[a] (0.2–0.7)	0.5 ± 0.03[a] (0.3–0.7) ±	0.1 ± 0.001[b] (0.07–0.16) ±	0.1 ± 0.01[b] (0.08–0.14)	0.27
Zn	1.8 ± 0.12[a] (1.2–2.7)	2.2 ± 0.23[a] (1.5–2.9)	0.7 ± 0.04[b] (0.5–1.0)	0.6 ± 0.12[b] (0.5–1.1)	1.32

[a.b.c.d]The averages shown by the different letters on each line are statistically different ($p < 0.05$).
X ± SE: Mean ± standard error. Number of samples sampled for each season: *N*: 20.

Heavy metals in fish tissues

The seasonal accumulation of heavy metals in various tissues (gill, liver and muscle) of pike fish are presented shown in Table 6. It has been observed that Al, Ba, Cr, Mn and Zn changed in the order gill>liver>muscle in all seasons. Changes in the amounts of Cu, Fe and Pb were liver>gill>muscle. When the seasons were examined in which the total heavy-metal levels of the pike fish were highest; Al and Ba in the winter; Cr, Mn, Pb and Zn in the spring; Cu and Fe in the summer.

Table 5. Seasonal distribution of heavy-metal concentrations in sediment.

Metals (mg/kg)	Seasons: Spring X± SE Min.–Max.	Summer X± SE Min.–Max.	Autumn X± SE Min.–Max.	Winter X± SE Min.–Max.	The annual average
Al	87.3 ± 10.72[**a**] (80.5–118.5)	105.3 ± 5.31[**a**] (80.6–125.1)	50.3 ± 1.32[**b**] (46.7–52.9)	48.9 ± 15.83[**b**] (22.3–74.9)	72.9
Ba	3.4 ± 0.07[a] (1.6–4.8)	3.3 ± 0.07[a] (1.8–4.4)	2.3 ± 0.06[b] (1.5–3.4)	2.3 ± 0.39[b] (1.5–3.4)	2.8
Cr	7.7 ± 1.24 (4.5–10.0)	8.6 ± 1.30 (5.5–12.7)	5.2 ± 1.12 (4.1–9.1)	6.3 ± 1.10 (4.1–9.3)	6.9
Cu	13.5 ± 1.62[a] (12.3–24.9)	14.6 ± 1.25[a] (13.0–25.7)	7.0 ± 1.19[b] (6.9–11.9)	8.1 ± 1.05[b] (6.8–11.5)	10.8
Fe	84.0 ± 10.6[a] (56.1–142.6)	108.2 ± 2.04[a] (74.2–133.1)	61.2 ± 1.52[b] (58.6–65.3)	80.3 ± 6.54[b] (69.8–128.4)	83.4
Mn	152.0 ± 10.14[a] (111.3–210.8)	157.7 ± 14.04[a] (110.5–201.7)	71.6 ± 10.12[b] (51.3–101.8)	69.7 ± 10.54[b] (48.0–104.4)	112.7
Pb	4.3 ± 0.33[a] (4.7–6.30)	5.1 ± 0.44[a] (4.4–6.13)	2.8 ± 0.26[b] (2.7–3.14)	3.1 ± 0.38[b] (2.6–3.13)	3.8
Zn	155.1 ± 16.42[a] (74.3–209.6)	166.2 ± 15.66[a] (65.7–240.3)	120.4 ± 12.15[b] (61.8–203.2)	113.1 ± 13.16[b] (38.6–195.5)	108.6

[**a.b.c.d**] The averages shown by the different letters on each line are statistically different ($p < 0.05$).
X ± SE: Mean ± standard error. Number of samples sampled for each seasons $N = 20$.

Table 6. Seasonal distribution of heavy-metal concentrations in the tissues of pike fish.

		Seasons			
Heavy metals (μg/g)	Tissues	Spring X± SE Min.–Max.	Summer X± SE Min.–Max.	Autumn X± SE Min.–Max.	Winter X± SE Min.–Max.
Al	Muscle	2.4 ± 1.10^{y} (0.8–6.6)	3.3 ± 1.34^{y} (1.2–5.8)	5.3 ± 0.13^{y} (5.2–5.5)	5.6 ± 1.60^{y} (3.1–8.5)
	Gill	68.0 ± 16.54^{xb} (22.0–138.9)	10.1 ± 0.60^{xb} (9.1–11.2)	19.8 ± 1.89^{xb} (16.3–22.8)	173.9 ± 24.28^{xa} (156.0–311.1)
	Liver	13.2 ± 2.26^{ya} (8.4–19.3)	2.7 ± 0.43^{yb} (2.1–3.6)	2.6 ± 0.31^{yb} (2.1–3.2)	4.9 ± 0.07^{yb} (4.8–5.0)
Ba	Muscle	0.6 ± 0.17^{zc} (0.5–0.8)	1.8 ± 0.15^{yb} (1.5–2.1)	0.3 ± 0.09^{yc} (0.2–0.5)	2.5 ± 0.22^{ya} (2.1–2.9)
	Gill	4.7 ± 0.59^{xab} (2.8–5.9)	6.0 ± 1.05^{xa} (4.1–7.7)	3.0 ± 0.37^{xb} (2.3–3.6)	6.3 ± 0.72^{xa} (4.8–7.2)
	Liver	2.4 ± 0.59^{y} (1.2–3.7)	1.2 ± 0.10^{y} (1.0–1.4)	1.2 ± 0.17^{y} (0.9–1.5)	1.4 ± 0.01^{y} (1.3–1.4)
Cr	Muscle	1.0 ± 0.27^{z} (0.6–2.0)	0.7 ± 0.12^{y} (0.5–0.9)	1.2 ± 0.29 (0.8–1.5)	1.2 ± 0.19^{y} (0.8–1.6)
	Gill	3.7 ± 0.33^{ya} (2.5–4.5)	2.1 ± 0.28^{xb} (1.6–2.6)	1.9 ± 0.04^{b} (1.08–2.0)	2.5 ± 0.06^{xb} (2.3–2.6)
	Liver	5.3 ± 0.60^{xa} (4.3–7.0)	0.5 ± 0.01^{yb} (0.4–0.5)	1.3 ± 0.25^{b} (1.0–1.8)	0.8 ± 0.01^{yb} (0.8–0.9)
Cu	Muscle	0.7 ± 0.08^{yb} (0.5–1.0)	0.6 ± 0.14^{yb} (0.4–0.9)	0.9 ± 0.10^{yb} (0.8–1.0)	2.1 ± 0.13^{ya} (1.8–2.3)
	Gill	0.7 ± 0.06^{yb} (0.5–0.9)	0.8 ± 0.06^{yb} (0.6–0.9)	0.8 ± 0.09^{yb} (0.6–1.0)	1.6 ± 0.08^{ya} (1.4–1.7)
	Liver	24.0 ± 4.9^{xab} (14.3–37.2)	37.7 ± 6.03^{xa} (26.6–47.4)	27.9 ± 4.72^{xa} (19.3–35.4)	10.1 ± 0.17^{xb} (9.9–10.5)
Fe	Muscle	11.3 ± 2.03^{zb} (6.7–18.8)	5.9 ± 0.85^{yb} (4.3–7.1)	11.4 ± 0.21^{yb} (11.1–11.7)	27.7 ± 5.98^{za} (16.8–37.4)
	Gill	184.7 ± 11.04^{ya} (148.7–216.1)	80.6 ± 9.15^{yb} (67.2–98.1)	109.1 ± 7.72^{yb} (98.2–124.0)	223.5 ± 17.08^{xa} (194.2–.253.4)
	Liver	388.0 ± 32.75^{xb} (353.8–486.2)	649.0 ± 25.42^{xa} (482.2–774.7)	332.0 ± 42.79^{xbc} (256.9–442.6)	102.5 ± 0.38^{yc} (101.8–103.2)
Mn	Muscle	1.8 ± 0.47^{y} (0.6–3.0)	1.2 ± 0.38^{y} (0.7–2.0)	1.7 ± 0.20^{z} (1.5–1.9)	1.2 ± 0.55^{y} (1.0–2.9)
	Gill	43.4 ± 4.06^{xa} (30.0–54.8)	28.3 ± 6.54^{xab} (19.4–41.1)	20.2 ± 1.28^{xb} (18.6–22.8)	30.9 ± 5.57^{xab} (20.1–38.8)
	Liver	5.2 ± 0.81^{yb} (3.3–7.0)	2.3 ± 0.28^{yc} (1.7–2.7)	7.4 ± 0.77^{ya} (5.9–8.4)	4.1 ± 0.05^{ybc} (4.0–4.3)
Pb	Muscle	0.8 ± 0.03^{b} (0.7–0.9)	0.6 ± 0.07^{yb} (0.5–0.8)	0.9 ± 0.01^{b} (0.8–0.9)	2.4 ± 0.21^{xa} (2.1–2.8)
	Gill	2.0 ± 0.71 (0.6–4.6)	0.6 ± 0.28^{y} (0.2–1.2)	0.8 ± 0.04 (0.7–0.9)	2.4 ± 0.11^{x} (2.2–2.6)
	Liver	0.8 ± 0.05 (0.7–0.9)	2.2 ± 0.63^{x} (1.1–3.3)	1.5 ± 0.90 (0.3–3.2)	0.5 ± 0.01^{y} (0.5–0.6)
Zn	Muscle	49.1 ± 3.03^{z} (40.8–57.9)	33.9 ± 7.3^{z} (25.4–48.4)	38.9 ± 6.75^{z} (32.1–45.6)	46.6 ± 4.71^{y} (40.3–55.8)
	Gill	930.0 ± 54.8^{xa} (787.8–1155.9)	717.0 ± 20.86^{xb} (691.6–758.4)	580.0 ± 38.43^{xb} (503.5–625.0)	738.9 ± 72.73^{xb} (614.8–866.7)
	Liver	320.2 ± 38.87^{ya} (250.3–431.3)	172.1 ± 15.69^{yb} (142.4–195.8)	301.8 ± 9.84^{ya} (287.8–320.8)	136.6 ± 1.12^{yb} (134.6–138.5)

$^{a.b.c.d}$ The averages shown by the different letters on each line are statistically different ($p < 0.05$).
$^{x.y.z}$ The averages shown by the different letters on each column are statistically different ($p < 0.05$).
X ± SE: Mean ± standard error, number of samples sampled for each season, respectively, $N = 21, 24, 20, 25$.

The ratios of heavy metals to tissues are given in Table 7. It was determined that the highest accumulation of Al, Ba, Mn, Pb and Zn in the gill tissue and Cr, Cu and Fe in the liver tissue (Table 7).

Table 7. The ratio of metal concentration in liver (L) and gill (G) to muscle (M) tissue of pike fish.

Heavy metals	Tissue ratio	Pike fish
Al	L:M	5.4
	G:M	28.3
Ba	L:M	4.0
	G:M	7.8
Cr	L:M	5.3
	G:M	3.7
Cu	L:M	34.2
	G:M	1.02
Fe	L:M	34.3
	G:M	14.1
Mn	L:M	2.8
	G:M	24.1
Pb	L:M	1.0
	G:M	2.5
Zn	L:M	6.5
	G:M	18.9

A total of 90 fish were sampled.

Seasonal BCF values of heavy metals are given in Table 8. In all seasons it was observed that the water played more significant role in heavy-metal accumulation in gill, liver and muscle tissues of pike fish than the sediment. Gill tissue; BCF ratios (gill/water and gill/sediment) in all heavy metals were highest in the winter and statistically significant ($p < 0.05$). Liver BCF ratios (liver/water); only Al level was highest in the winter whereas Cu, Fe, Pb and Zn were highest in the autumn, Cr and Mn were highest in the spring and Ba in the summer ($p < 0.05$). When the BCF ratios (liver/sediment) were examined in the liver, Ba, Cu, Mn, Pb, and Zn were found to be highest in the autumn, Al in winter, Cr in spring and Fe in summer ($p < 0.05$).

In muscle tissues, BCF (muscle/water) ratios of all heavy metals were found to be highest in winter and autumn ($p < 0.05$) and BCF (muscle/sediment) ratios of all heavy metals were found to be highest in the autumn and winter ($p < 0.05$), except for Mn.

The seasonal distribution of serum biochemical parameters of pike *Esox lucius* (Linnaeus, 1758) fish is given in Table 9. It was determined that the serum cortisol was in the order of summer>spring>autumn = winter. Serum total protein and albumin levels were determined as summer>autumn>winter>spring. Glucose, cholesterol and triglyceride levels were found to be in the order of summer>spring>autumn = winter. The alanine aminotransferase, aspartate aminotransferase, lactate dehydrogenase and alkaline phosphatase levels were identified as spring>winter>summer>autumn. Calcium, phosphorus and iron levels were determined as summer>autumn>winter>spring, sodium, potassium and chloride levels were observed as spring>summer>autumn = winter and these findings were regarded as significant ($p < 0.05$).

Haematological parameters of pike fish

There was a significant difference in total leukocyte-erythrocyte-platelet counts, granulocyte, agranulocyte ratios, haemoglobin amount and haematocrit values to the extent of the seasons in pike fish ($p < 0.05$). These significant differences; total leukocyte-erythrocyte-platelet count, haemoglobin amount, haematocrit value and granulocyte

Table 8. Seasonal concentrations (μg g^{-1}wt) and bioconcentrations factor (BCF) during spring, summer, autumn, winter of metals in different tissues of pike fish.

		Al		Ba		Cr		Cu		Fe		Mn		Pb		Zn	
Fish tissue	Seasons	W	S	W	S	W	S	W	S	W	S	W	S	W	S	W	S
Gill	Spring	136.0^{c}	0.70^{c}	117.5^{b}	1.38^{c}	92.5^{b}	0.48^{a}	1.4^{c}	0.05^{c}	16.3^{b}	2.19^{b}	48.2^{c}	0.28^{b}	5.0^{b}	0.46^{b}	516.6^{c}	5.99^{b}
	Summer	16.8^{d}	0.09^{d}	85.7^{d}	1.81^{b}	42.0^{c}	0.24^{d}	0.8^{d}	0.05^{c}	4.6^{d}	0.74^{d}	25.7^{d}	0.17^{c}	1.2^{d}	0.11^{d}	325.9^{d}	4.31^{c}
	Autumn	198.0^{b}	0.39^{b}	100.0^{c}	1.30^{c}	95.0^{b}	0.36^{c}	2.0^{b}	0.11^{b}	14.9^{c}	1.78^{c}	101.0^{b}	0.28^{b}	8.0^{a}	0.28^{c}	828.5^{b}	4.81^{c}
	Winter	1739.0^{a}	3.55^{a}	210.0^{a}	2.73^{a}	125.0^{a}	0.39^{b}	5.3^{a}	0.19^{a}	37.2^{a}	2.78^{a}	154.5^{a}	0.44^{a}	2.4^{c}	0.77^{a}	1232.5^{a}	6.53^{a}
Liver	Spring	26.4^{b}	0.15^{a}	60.0^{b}	0.70^{b}	132.5^{a}	0.68^{a}	48.0^{b}	1.77^{c}	34.3^{c}	4.61^{b}	48.2^{a}	0.03^{b}	2.0^{c}	0.18^{c}	177.8^{c}	2.06^{a}
	Summer	4.5^{c}	0.02^{d}	85.7^{a}	0.36^{c}	42.0^{b}	0.05^{d}	37.7^{c}	2.58^{b}	37.2^{b}	5.99^{a}	2.09^{d}	0.01^{c}	4.4^{b}	0.43^{b}	78.2^{d}	1.03^{b}
	Autumn	26.0^{b}	0.05^{c}	40.0^{d}	1.30^{a}	43.3^{b}	0.25^{b}	69.7^{a}	3.98^{a}	45.3^{a}	5.42^{a}	37.0^{b}	0.10^{a}	15.0^{a}	0.53^{a}	431.1^{a}	2.50^{a}
	Winter	49.0^{a}	0.10^{b}	46.6^{c}	0.60^{b}	40.0^{b}	0.12^{c}	33.6^{d}	1.24^{d}	17.0^{d}	1.27^{d}	20.5^{c}	0.05^{b}	5.0^{b}	0.16^{c}	227.6^{b}	1.20^{b}
Muscle	Spring	4.8^{b}	0.02^{b}	15.0^{c}	0.17^{c}	25.0^{b}	0.12^{b}	1.4^{b}	0.05^{c}	1.0^{c}	0.13^{b}	2.0^{b}	0.01^{b}	2.0^{c}	0.18^{c}	27.2^{c}	0.31^{b}
	Summer	5.5^{a}	0.03^{b}	25.7^{b}	0.54^{b}	14.0^{c}	0.08^{b}	0.6^{c}	0.04^{c}	0.3^{d}	0.05^{c}	1.1^{c}	0.07^{a}	1.1^{d}	0.11^{d}	15.4^{d}	0.20^{c}
	Autumn	5.3^{a}	0.10^{a}	10.0^{d}	0.13^{d}	60.0^{a}	0.23^{a}	2.2^{a}	0.12^{b}	1.5^{b}	0.18^{b}	8.5^{a}	0.02^{b}	9.0^{a}	0.32^{b}	55.5^{b}	0.32^{b}
	Winter	5.6^{a}	0.11^{a}	83.3^{a}	1.08^{a}	60.0^{a}	0.19^{a}	0.7^{c}	0.25^{a}	3.9^{a}	0.34^{a}	0.6^{d}	0.01^{b}	2.4^{b}	0.77^{a}	77.6^{a}	0.41^{a}

Water (W), sediment (S).
A total of 90 fish were sampled.
[a.b.c.d] It shows the statistical difference for each tissue between the seasons ($p < 0.05$).

Table 9. Seasonal distribution of serum biochemical parameters of pike fish.

Serum biochemical parameters	Seasons			
	Spring	Summer	Autumn	Winter
Metabolites				
Total protein (mg/dL)	1.9 ± 0.49^c	3.6 ± 0.21^a	3.0 ± 0.17^b	2.4 ± 0.12^b
Albumin (mg/L)	0.8 ± 0.11^c	2.0 ± 0.07^a	1.6 ± 0.07^b	1.1 ± 0.08^b
Glucose (mg/L)	126.8 ± 5.29^b	149.4 ± 6.16^a	103.8 ± 4.31^c	90.2 ± 2.77^c
Cholesterol (mg/L)	213.4 ± 5.09^a	238.0 ± 4.04^b	204.0 ± 4.73^c	202.1 ± 4.74^c
Triglyceride (mg/L)	112.2 ± 6.14^b	166.2 ± 8.56^a	94.2 ± 5.58^c	89.2 ± 6.32^c
Blood urea nitrogen (mg/L)	5.8 ± 0.48^b	7.4 ± 0.43^a	3.4 ± 0.66^c	3.1 ± 0.54^c
Hormone				
Cortisol	825.5 ± 22.16^b	897.7 ± 28.23^a	413.6 ± 18.96^c	377.4 ± 16.71^c
Enzymes				
Aspartate amino transferase (AST) (U/L)	1148.4 ± 36.36^a	916.0 ± 22.36^b	749.5 ± 28.43^d	1045.3 ± 26.92^c
Alanine amino transferase (ALT) (U/L)	39.0 ± 3.67^a	24.6 ± 2.62^c	25.2 ± 2.43^c	34.2 ± 2.52^b
Lactate dehydrogenase (LDH) (U/L)	1553.8 ± 22.13^a	1336.2 ± 23.96^a	1097.8 ± 21.55^c	1465.3 ± 24.82^b
Alkaline phosphatase (ALP) (U/L)	61.4 ± 2.10^a	50.8 ± 2.26^c	48.8 ± 2.84^c	58.2 ± 2.82^b
Electrolytes				
Calcium (mg/L)	8.4 ± 0.24^c	16.9 ± 0.75^a	14.6 ± 0.80^a	10.3 ± 0.77^b
Phosphorus (mg/L)	13.5 ± 0.68^b	24.9 ± 1.87^a	19.1 ± 1.12^a	18.2 ± 1.43^a
Iron (mg/L)	90.0 ± 6.55^c	139.2 ± 5.67^a	120.5 ± 5.12^a	102.3 ± 4.21^b
Sodium (nmol/L)	146.2 ± 4.92^a	127.0 ± 3.44^b	110.8 ± 2.42^c	112.1 ± 4.49^c
Potassium (nmol/L)	5.7 ± 0.95^a	2.84 ± 0.29^b	1.45 ± 0.28^c	1.66 ± 0.23^c
Chloride (nmol/L)	96.2 ± 3.98^a	87.3 ± 3.44^b	82.2 ± 2.03^c	79.2 ± 3.21^c

$^{a.b.c.d}$The averages shown by the different letters on each line are statistically different ($p < 0.05$).
X ± SE: Mean ± standard error, number of samples sampled for each season, respectively; N = 21, 24, 20, 25.

ratio increased from spring to summer, decreased from autumn to winter. On the contrary agranulocyte ratios increased from autumn to winter and decreased from spring to summer (Table 10).

Histopathology of pike fish

The liver and gill tissues of pike fish *Esox lucius* (Linnaeus, 1758) in Ladik Lake were examined histopathologically, and the lesion types in these tissues shown seasonally in Table 11 (liver) and in Table 12 (gill).

Although there were not many toxicological lesions that can be caused by toxic substances when considering liver tissues, the most common findings were; vacuolization in hepatocytes, small bleeding zones (petechiae), mononuclear cell infiltration and hepatocellular degeneration (Table 11 and Figure 2).

Table 10. Seasonal distribution of total leukocyte-erythrocyte-thrombocyte counts, granulocyte-agranulocyte rates, haemoglobin count and haematocrit values of pike fish.

Blood parameters	Seasons			
	Spring	Summer	Autumn	Winter
Total leukocyte count 10^3/mm^3	8.3 ± 0.11^b	9.6 ± 0.14^a	7.1 ± 0.17^c	6.8 ± 0.14^d
Granulocyte ratio (%)	80.3 ± 1.65^b	86.2 ± 1.22^a	76.2 ± 1.85^c	70.2 ± 1.14^d
Agranulocyte ratio (%)	20.7 ± 1.65	13.8 ± 1.22	23.8 ± 1.85	29.8 ± 1.14
Number of erythrocytes 10^3/mm^3	2.48 ± 0.39^b	2.90 ± 0.46^a	1.75 ± 0.17^c	1.35 ± 0.12^d
Amount of haemoglobin (g/dL)	9.4 ± 0.68^b	11.3 ± 0.45^a	8.2 ± 0.72^c	7.5 ± 0.14^d
Haematocrit value ratio (%)	42.7 ± 0.74^b	46.8 ± 0.81^a	32.8 ± 0.74^c	23.8 ± 0.70^d
Number of platelets (10^3/mm^3)	370.2 ± 7.55^b	402.1 ± 7.95^a	315.2 ± 8.10^c	214.5 ± 6.21^d

$^{a.b.c.d}$ The averages shown by the different letters on each column are statistically different from each other ($p < 0.05$).
Mean ± standard error, number of samples sampled for each season, respectively; N = 21, 24, 20, 25.

Table 11. Distribution of the lesions seen in the liver tissues of the pike fish according to the seasons.

Toxicopathological lesions observed in liver tissue	Spring	Summer	Autumn	Winter
1- Vacuolization in hepatocytes	++	++++	+++	+
2- Bleeding within the tissue zones (petechiae)	+	+++	++	
3- Mononuclear cell infiltration	+	+++	++	
4- Hepatocellular degeneration	++	++++	+++	+
5- Fatty liver	+			

+Lesion rate in liver tissues.
Number of samples sampled for each season, respectively; N = 21, 24, 20, 25.

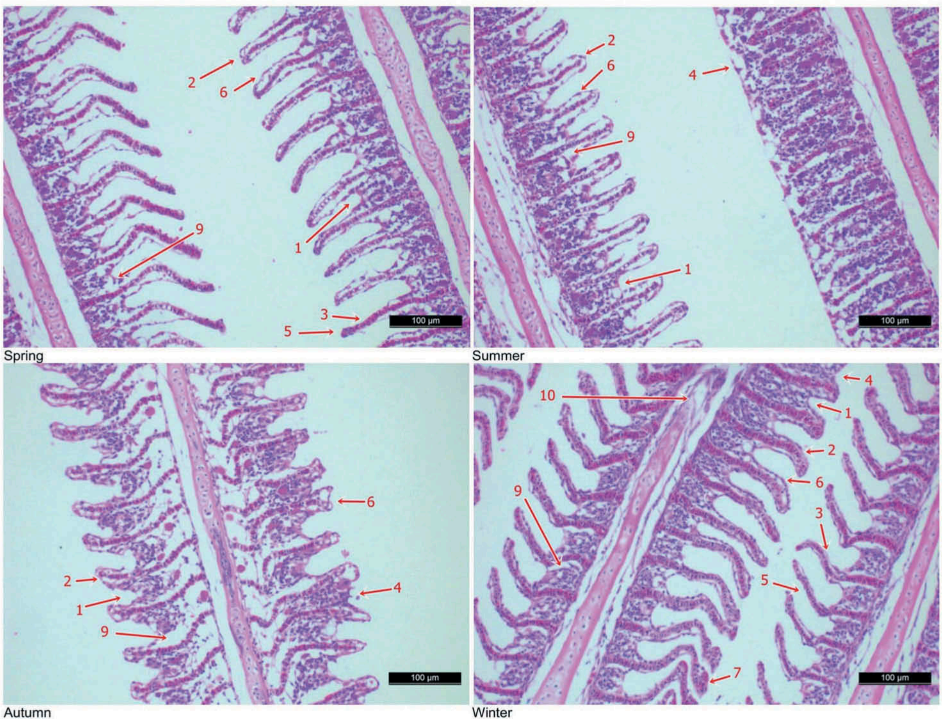

Figure 2. Histopathological changes of pike fish liver tissue in different season.
(Arrows 1 – vacuolization in hepatocytes; 2 – bleeding within the tissue zones (petechiae); 3 – mononuclear cell infiltration) HE X200.

During the spring period, liver tissues were observed to be fatty because of the tendency of some pike fish to increase their nutritional indices that may be because of the reproductive behaviour.

When the gill tissues are taken into consideration, the most common findings from toxicological lesions that may be because of the adverse environmental conditions are; reduction in the space between the secondary lamellae, fusion, merger, thickening, swelling and shortening of secondary lamellae. Furthermore, vacuolization in the gill epithelium and cartilage damage were detected. The severity of these lesions was in the order of summer>autumn>spring>winter (Table 12 and Figure 3).

Table 12. Distribution of the lesions seen in the gill of pike fish (*Esox lucius* Linnaeus, 1758) in Ladik Lake (Samsun, Turkey) according to the seasons.

Toxicopathologic lesions observed in the gill	Spring	Summer	Autumn	Winter
1- Reduction in the space between the secondary lamellae	+	+++	++	
2- Shortening in secondary lamellae	+	+++	++	
3- Fusion of secondary lamellae	+	+++	++	
4- Merger in the secondary lamellae	+	+++	++	
5- Thinning in secondary lamellae	++	++++	+++	+
6- Thickening of the secondary lamellae	+	+++	++	
7- Swelling in the secondary lamellae	++	++++	+++	+
8- Curling of secondary lamellae	++	++++	+++	+
9- Vacuolization in the epithelium	++	++++	+++	+
10- Cartilage tissue damage		++	+	

+Lesion rate in gill tissue.
Number of samples sampled for each season, respectively; N = 21, 24, 20, 25.

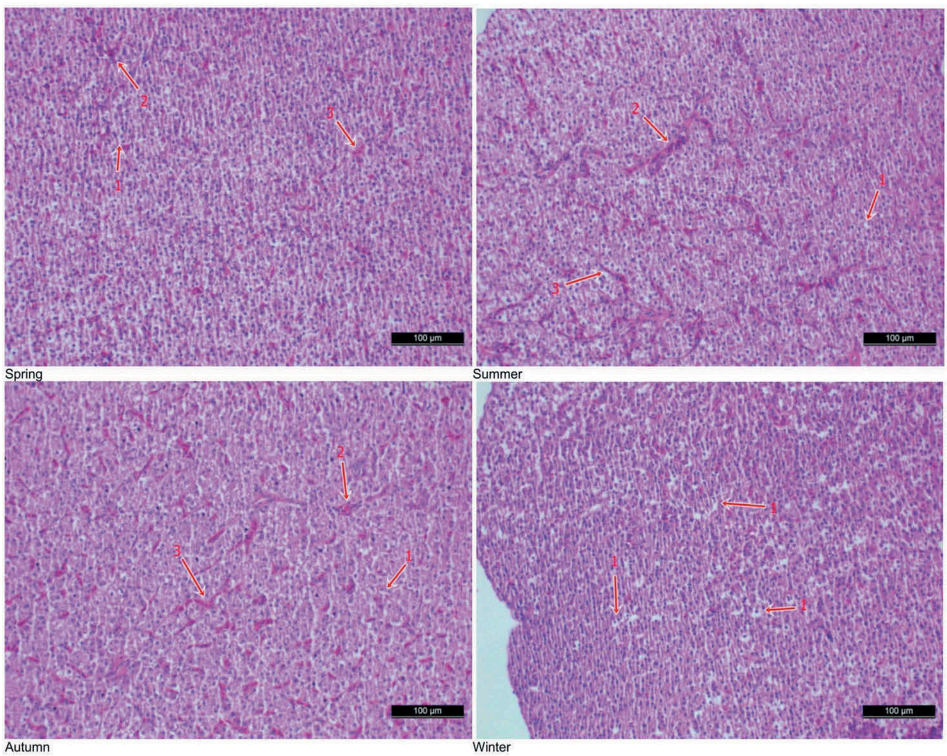

Figure 3. Histopathological changes of pike fish (*Esox lucius* Linnaeus, 1758) gill tissue in different season. (Arrows 1 – reduction in the space between the secondary lamellae; 2 – shortening in secondary lamellae; 3 – fusion of secondary lamellae; 4 – merger in the secondary lamellae; 5 – thinning in secondary lamellae; 6 – thickening of the secondary lamellae; 7 – swelling in the secondary lamellae; 9 – vacuolization in the epithelium; 10 – cartilage tissue damage) HE X100.

Discussion

Heavy metal in water and sediment

In Ladik Lake, it was observed that Fe was the most common heavy metal in all seasons. It was understood that the annual average values of Cr, Mn and Pb were above the WHO standards and the water quality was close to Class III according to TEG (Table 13).

Table 13. Comparison of heavy metals measured in water samples in Ladik Lake (Samsun, Turkey) literature values.

Locations	Heavy metals μgL^{-1}								References
	Al	Ba	Cr	Cu	Fe	Mn	Pb	Zn	
Ladik Lake, Turkey	320	40	300	550	10500	600	270	1320	Present study
	200	–	50	20	300	500	10	3000	WHO (2004)
TEG[a]									
Class I	–	–	20	20	300	100	10	200	Turkish Environmental Guidelines (1988)
Class II	–	–	50	50	1000	500	20	500	
Class III	–	–	200	200	5000	3000	50	2000	
Class IV	–	–	>200	>200	>5000	>3000	>50	>2000	
Tigris River, Turkey	–	–	<5	165	388	467	0.342	37	Varol and Şen (2012)
Sakarya River, Turkey	–	–	228	4362	–	–	5789	1360	Dundar and Altundag (2007)
Challawa River, Nigeria	–	–	924	390	5668	1681	840	2227	Dan'Azumi and Bichi (2010)
Beyşehir Lake, Turkey	–	–	–	100	100–2740	–	–	20–240	Tekin-Özan and Kir (2008)
Isikli Lake, Turkey	–	11	4	7.5	360	50	9.2	440	Tekin-Ozan and Aktan (2012)
Dianchi Lake, China	–	–	4	2	225	256	266	17	Wang et al. (2014)
Insko Lake, Poland	–	–	–	660	2180	850	–	1000	Rajkowska and Protasowicki (2011)
Wiola Lake, Poland	–	–	–	510	2970	2510	–	1710	
Upper lake, India	–	–	30	12	–	–	330	300	Malik et al. (2010)

Although the literature indicates that the concentration of Cr was higher in rivers than in lakes (Dundar and Altundag 2007; Dan'Azumi and Bichi 2010), the amount of Cr in Ladik Lake was found at low level in comparison with some studies (Rajkowska and Protasowicki 2011) and at the same time it was above the standards (Turkish Environmental Guidelines 1988; WHO 2004). It can be assumed that the Cr accumulation above the standard in Lake Ladik that caused by cesspool/sewage wastes and sewers.

The research was carried out by Rajkowska and Protasowicki (2011), which demonstrated the high Cu content in the lakes of Insko and Wisola, is similar to our study. The abundance of copper in these lakes is an indication that agricultural chemicals and household wastes are discharged into those areas. The amount of Pb was found similar to Dianchi and Upper Lakes (Malik et al. 2010; Wang et al. 2014). The amount of Al in the Ladik Lake is above the standards and it is thought that the excess amount of chemicals was resulted from the wastes of the cement factory located near the lake (WHO 2004). The Mn level above the standards can be characterized by lasting human activities (domestic waste) and low water depth (Lee et al. 2009). The reason for the high level of heavy metals in Ladik Lake, which is characterized by very small water depths with floating islets, is domestic, industrial and agricultural activities (Karouna-Renier and Sparling 2001; Casey et al. 2007).

It was reported that Cd and Cu are first accumulating heavy metals in the sediments and in the bottoms of the lakes as a result of domestic, industrial and agricultural pollution discharged to lake (Dawson and Macklin 1998; Liang et al. 1999). Similarly, Cr and Cu were found to be high although the Cd in Ladik Lake could not be determined. Also, the amount of Cu was similar to the amount measured in Insko and Wisola Lake (Rajkowska and Protasowicki 2011; Benzer et al. 2013). The Fe concentration was found at low level in comparison with the amounts measured in Isikli, Mogan and Karatas Lakes, higher than the concentrations measured in Dianchi, Insko and Wisola Lakes. The low concentration of Mn in Ladik Lake indicates that the bottom mud accumulation was at low level and that household wastes were not accumulated too much. Pb concentration was higher than the concentration measured in the Mogan and Karataş Lakes (Başyiğit and Tekin-Özan 2013; Benzer et al. 2013), and significantly lower than the concentrations measured in Veerenam, Dangting, Dianchi, Karla and Manchar Lakes (Arain et al. 2008; Suresh et al. 2012; Li et al. 2013; Wang et al. 2014; Skordas et al. 2015) (Table 14).

Bioconcentration factor

It was reported that the accumulation of metal in fish changes according to the water, geologic structure of the environment, the type of feeding (herbivor–carnivor–omnivor) and the type of metal discharged into the water (Uysal et al. 2009; Subotić et al. 2013).

The fish muscle tissues generally consumed by human being. Fish accumulate more heavy metals in active metabolic tissues such as gills, liver and kidney than other tissues (Karadede et al. 2004; Tekin-Özan and Kir 2008). In this study the ratios of metals in liver (L), gill (G) and muscle (M) tissue in pike fish given in Table 7. Since the gill tissue is exposed to the outside environment, it is expected that the heavy-metal accumulation in these tissues would be more. In this study, it was determined that Al, Ba, Mn, Pb and Zn were accumulated more in the gill tissue. In addition, the liver of the fish was

Table 14. Comparison of heavy metals measured in sediment samples in Ladik Lake (Samsun, Turkey) with literature values.

	Heavy metals mg kg^{-1}								
Locations	Al	Ba	Cr	Cu	Fe	Mn	Pb	Zn	References
Ladik Lake, Turkey	72.9	2.8	6.9	10.8	83.4	112.7	3.8	108.6	Present study
Mogan Lake, Turkey	–	–	28.5	15.3	3577	125.6	0.82	13.7	Benzer et al. (2013)
Isikli Lake, Turkey	–	85.7	94.6	22.7	16807.9	505.9	–	70.8	Tekin-Özan and Aktan (2012)
Karataş Lake, Turkey	–	–	37.5	16.7	6968.4	227.2	0.88	28.1	Başyiğit and Tekin-Özan (2013)
Hazar Lake, Turkey	–	–	45	38	–	–	–	79	Özmen et al. (2004)
Veeranam Lake, India	–	–	88.6	94.1	–	–	30.0	180.0	Suresh et al. (2012)
Dangting Lake, China	–	–	88.2	47.8	–	–	60.9	185.2	Li et al. (2013)
Dianchi Lake, China	–	–	78	71	23.3	757	189	140	Wang et al. (2014)
Insko Lake, Poland	–	–	–	17.5	21.8	256	–	138.3	Rajkowska and Protasowicki (2011)
Wiola Lake, Poland	–	–	–	19.5	13.7	189	–	149.6	
Karla Lake, Grecee	–	–	298.8	38.3	–	–	34.3	31.2	Skordas et al. (2015)
Manchar Lake, Pakistan	–	–	20	21	–	–	18.9	96.6	Arain et al. (2008)

preferred more as an indicator of water pollution because of its metabolic role such as in storage, detoxification and distribution (Licata et al. 2005). It was determined that some heavy metals especially accumulate in this tissue. In this study, it was determined that heavy metals Cr, Cu and Fe were accumulated more in liver tissue (Table 7).

In this study, maximal BCF was observed in the gill tissue. It can be said that the gill tissue of the fish was directly in contact with the external environment, feeding of the fish buried under the sediment and housing activities of the fish. It was detected that the accumulation of Al, Ba, Cr, Mn and Zn were highest in the gill tissue, Cu, Fe, and Pb accumulation were the highest in the liver tissue (Table 8). The lowest ratio of BCF was determined as fish muscle tissue. When the accumulation of heavy metals was examined, it was determined that Zn > Al > Ba > Cr > Mn > Fe > Cu > Pb. Consequently, it was found out that these findings were similar to previous studies (Uluturhan and Kucuksezgin 2007; Rajkowska and Protasowicki 2013; Subotić et al. 2013; Salem et al. 2014; Wang et al. 2015).

Correlation

Heavy-metal screening activities in an aquatic environment are extremely important both for animal health and for human health (Dural et al. 2006). The heavy-metal accumulation in the aquatic environment is greater in the sediment. Heavy metals pass to water and organisms through redox and biological reactions from the sediment. This transition is mostly through either gill or through nutrition (Ptashynski et al. 2002).

In our study, it was found statistically significant that there is a high positive correlation between water and sediment in other heavy-metal specimens except Ba ($p < 0.05$). In addition, when the correlation coefficients between water and fish tissues are compared; Cu and Fe have positive correlation between water and liver tissue and Mn has negative correlation, Fe has positive correlation when compared with water and gill, and the negative correlation of Cr between water and muscle tissue was statistically significant ($p < 0.05$) (Table 15). Results in our study are similar to previous studies (Petkovšek et al. 2012; Rajkowska and Protasowicki 2013).

Table 15. Correlation coefficients of water, sediment and fish tissue (liver, gill and muscle) of pike fish.

Heavy metals		Water	Sediment	Liver	Gill	Muscle
Al	Water	1	0.994*	0.345	−0.488	−0.920
	Sediment		1	0.238	−0.529	−0.873
	Liver			1	0.211	−0.656
	Gill				1	0.422
	Muscle					1
Ba	Water	1	0.717	−0.169	0.433	0.222
	Sediment		1	0.559	0.245	0.144
	Liver			1	−0.039	−0.338
	Gill				1	0.920*
	Muscle					1
Cr	Water	1	0.954*	0.221	0.262	−0.964*
	Sediment		1	0.199	0.355	−0.916*
	Liver			1	0.920*	0.046
	Gill				1	0.009
	Muscle					1
Cu	Water	1	0.794**	0.849*	−0.499	−0.656
	Sediment		1	0.572	−0.508	−0.620
	Liver			1	−0.818**	−0.899**
	Gill				1	0.981*
	Muscle					1
Fe	Water	1	0.864*	0.947*	0.642*	−0.769
	Sediment		1	0.662**	−0.267	−0.343
	Liver			1	−0.828	−0.924
	Gill				1	0.845**
	Muscle					1
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	Liver			1	−0.828	−0.924
	Gill				1	0.845**
	Muscle					1
Mn	Water	1	0.922*	−0.630	0.497	−0.044
	Sediment		1	−0.552	0.576	−0.069
	Liver			1	−0.266	0.779*
	Gill				1	0.257
	Muscle					1
Pb	Water	1	0.987*	0.522	−0.320	−0.670
	Sediment		1	0.528	−0.298	−0.573
	Liver			1	−0.957*	−0.725
	Gill				1	0.741
	Muscle					1
Zn	Water	1	0.998*	0.070	0.498	−0.306
	Sediment		1	0.134	0.490	−0.306
	Liver			1	0.183	0.237
	Gill				1	0.669
	Muscle					1

* Significant level of test $p < 0.05$.
** Significant level of test $p < 0.01$.
A total of 80 samples were compared for water and sediment.
A total of 90 samples were compared for the tissues.

Heavy metal in fish tissue

According to the heavy-metal results of the studied fish tissues, levels of Cr, Cu, Pb and Zn were found to exceed the permissible limits according to FAO 1983, EU Commission

Regulation and TFC in various seasons and tissues. However, the results of Fe and Mn were found to be higher than the literature results (Türkmen et al. 2006; Sivaperumal et al. 2007; Türkmen and Ciminli 2007).

Biochemistry

It is reported that the seasonal variation of blood and serum biochemical content of fish changed depending on nutrition, reproduction and environment characteristics (Lenhardt 1992; Levesque et al. 2002; Aras et al. 2008). According to Lenhardt (1992), it was reported that the total protein and albumin content of pike fish was at the lowest level in spring when compared to the seasons and it was originated from the breeding cycle. Guijarro et al. (2003) found triglyceride, cholesterol, HDL and VLDL levels highest in May and June that was spawning period in *Tinca tinca* fish species. Besides, it was reported that triglyceride and cholesterol levels in the sea bass fish decreased during the winter (De Pedro et al. 2005). Growth, intermedia metabolism and enzyme activities in perch fish exposed to Cd, Zn and Cu contamination because of mining activities were investigated. In the case of reference fish (control group), the levels of liver glycogen and triglyceride in the summer was higher than in the autumn, while the seasonal structure in fish taken from the contaminating region showed a difference. AST levels in fish in contaminated areas were higher in the autumn but not higher in summer. Researchers reported that the growth, seasonal hepatic glycogen and triglyceride cycles and metabolic enzyme activities of fish exposed to heavy metal at sublethal doses were affected (Levesque et al. 2002). Therefore, it can be said that the results of our research in pike fish are similar to those of the literature mentioned above.

Haematology

It was reported that haemoglobin and haematocrit values in the sea bass fish were similar in the spring and summer seasons, total leukocyte counts in female fish were significantly higher in summer than in other seasons (De Pedro et al. 2005), in spring and summer RBC and haematocrit values changed significantly in male fish compared to autumn and winter, and WBC levels in the spring and the winter declined significantly when compared to summer and autumn (Collazos et al. 1998). RBC, Hct, MCV and WBC values were highest in May and lowest in winter. The highest values for Hb, MCH and MCHC were in January while the lowest values were occurred in the spring. As a result; investigators reported that the parameters of this study were affected by many internal and external factors such as reproductive cycle, water temperature and metabolic rate (Aras et al. 2008). The seasonal variation of the blood parameters of pike fish increased in the spring and summer and decreased in the autumn and the winter. These data are similar to the literature (Collazos et al. 1998; Guijarro et al. 2003).

Histopathology

Gills are organs responsible for biological activities such as osmoregulation and excretion in the fish and are subject to rapid changes or damage in the face of adverse physical and chemical effects of the external environment (Fernandes and Mazon 2003).

In adverse environmental conditions, it was reported that the histopathological findings frequently observed in the gill tissue of fish were changes (thickening, flattening, shortening, hyperplasia, vacuolization in the epithelium and hypertrophy etc.) in the secondary lamellae (Ribeiro et al. 2000; Cerqueira and Fernandes 2002; Martinez et al. 2004). In our study, histopathology was similar to the lesions mentioned in the literature had been seen. These histopathologic lesions were more common in the summer. However, it was concluded that the reason why it was seen with different intensity in all seasons was that water pollutants have been continuously discharged in all seasons and may have been caused by the increase and decrease of pollutant constants because of the decrease and proliferation of water in different seasons (Uçar and Atamanalp 2009).

Because of its function, position and blood supply liver is the organ most associated with the detoxification and biotransformation process. It is also one of the organs most affected by water contamination (Camargo and Martinez 2007). It has been reported that histopathological lesions in the liver are caused by organophosphate compounds in the structure of agricultural pollutants (Fanta et al. 2003).

Vacuolization, hemosiderin and fibrosis were observed in the liver of carp fish exposed to the solution prepared by taking 1.25 mg/L of Cd + Pb + Cr + Ni heavy metals (Vinodhini and Narayanan 2008). Benedetti et al. (1981) reported cytoplasmic vacuolization of hepatocytes of *Ictaburus nebulous* liver because of copper contamination. Kumar and Pant (1981) investigated the vacuolization in and out of liver hepatocytes, necrotic changes in liver and induction of vacuolization of *Puntius conchonius* exposed to copper and zinc. Dalela et al. (1984) reported that exposure of lethal and sublethal concentrations of copper and cadmium to *Cyprinus carpio* caused necrosis, hypertrophy and atrophy of liver tissues, deterioration of liver cells, and formation of spaces between the disruptive tissues of cells. In addition, Deore and Wagh (2012) observed vacuolization in the cytoplasm, degeneration in nuclei, swellings, nuclear pyknosis, necrosis and deterioration of shapes in the liver hepatocytes histopathology of *Channa gachua* (Ham) freshwater fish that was exposed to lethal (1.0625 ppm, 1.4202 ppm) and sublethal (0.1062 ppm, 0.0531 ppm, 0.1420 ppm and 0.0710 ppm) mercury chloride and copper chloride concentrations. Mohamed (2009) reported that the liver of *Tilapia zillii* and *Solea vulgaris* from Lake Qarun – Egypt showed vacuolar degeneration in the hepatocytes, focal areas of necrosis, thrombosis formation in central veins, dilation and congestion in blood sinusoids and fibrosis. Additionally, liver of *Oreochromis niloticus* from polluted wetland environments in Saudi Arabia showed vacuolization of the hepatocytes, sinusoidal congestion, necrosis of the parenchyma tissue, nuclear pyknosis, eosinophilic hepatocellular degeneration, pigment accumulation, an increase in the number and size of melanomacrophage centres (Abdel-Moneim et al. 2012).

Generally histopathological findings in the liver were determined as an increase in vacuoles in hepatocyte cytoplasm, enlargement in lysosomes, changes in cell shape, necrosis, ischemia (temporary anaemia of a region) and fat degeneration.

Conclusion

In this study, the pollution of Ladik Lake and the health status of pike fish, an economically important species, exposed to pollution in Ladik Lake were evaluated seasonally for a whole year. It has been determined that pike fish which is frequently preferred for human nutrition and Lake need to be monitored in terms of heavy metals such as Cr, Cu, Pb and Zn. It has been concluded that it is significant to report environmental factors (e.g. nutrition, tourism, sports, etc.) that may threat human health and protect the wild life. Moreover, it is crucial to state that inventory information must be gathered on the purpose of sustainable environmental policies.

Disclosure statement

No potential conflict of interest was reported by the authors.

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