Heavy Metal Accumulation in Lake Sediments in the Impact Zone of Trout Cage Farm

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ABSTRACT

Chemical residues from aquaculture, especially heavy metals, are harmful to environment. The present study assessed the seasonal dynamics and potential toxic effects of heavy metal accumulation (Cr, Cu, Zn, and Pb) in lake sediments within the impact zone of the trout cage farm (Lake Ladoga, Russia). The tasks included the layer-by-layer analysis of total content of the heavy metals and their labile forms in sediment cores collected in the winter and autumn seasons. Four sites located directly next to the cages and three reference sites (150, 600, and 1,000 m from the farm) were studied. Sediments cores collected using gravity corer were analyzed for total content analysis, where sediment samples were decomposed with strong acids ($HClO_4$, HF, HCl, and HNO_3), and the labile forms were extracted by ammonium acetate. The results showed that sediments under fish cages were significantly polluted by Zn and Cu especially towards the end of the active feeding period (autumn), thus potentially increasing sediment toxicity. Therefore, Zn and Cu can be utilized as geochemical markers for assessing the thickness of the accumulated contaminated sediment layer in the impact zone of cage farms. Pollution of the sediments by heavy metals is local and limited to the studied bay. Hence, there is no risk of secondary pollution of the water column according to the RAC index. However, the absolute content ($mg \cdot kg^{-1}$) of labile Zn is much higher under the cage than at reference sites.

Keywords: Environmental pollution, Fish farm impacts, RAC index, Sediment toxicity

INTRODUCTION

Aquaculture, particularly cage farming, has been widely used to increase fish production around the world. However, this practice often leads to serious environmental problems. Fish farming operations contribute a substantial number of wastes, including uneaten feed and excretory products, to the aquatic ecosystem. As a result, feed, antibiotics, antifouling paints, and other pollutants, including heavy metals, accumulate in sediments around the cages (Sapkota *et al*., 2008; Liang *et al*., 2016). It has been reported Cr, Cu, and Zn are major component found in sediments due to fish feed promoting fish growth, but Cu and Pb are also widely used in antifouling coatings for cage rafts (Liang *et al*., 2016; Zhang *et al*., 2018). The chemical composition of sediments provides a geochemical indicator of the pollution level. Additionally, heavy metals could accumulate in aquatic products along the food web of the ecosystem (Yu *et al*., 2012), including in cage fish (Xie *et al*., 2020). Therefore, biogeochemical study is necessary to assess environmental effects from cage farms and associated risks for water resources.

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This paper is the part of the scope of cage farm research and influence of cage culture on freshwater ecosystems (Ryzhkov *et al*., 2011; Bakhmet *et al*., 2017; Kaya and Pulatsu, 2017; Varol, 2019). It should be noted that eutrophication processes in lakes and the accumulation of wastes in their sediments can be accelerated due to low flushing rates. In addition, the high dilution rate and recycling process of cage fish farms induce minimal changes in water quality (Varol, 2019). Lake Ladoga is one of the leaders in rainbow trout farming production in the Russian Federation. Previous research predicted the negative impact of polluted sediments on cage fish and the aquatic ecosystem of Lake Ladoga in terms of organic matter input and the physicochemical parameters such as pH and Eh of water and sediments (Lapenkov *et al*., 2023). However, information on the dynamics of heavy metals in this context is limited. Therefore, this study was conducted with the aim of evaluating the seasonal dynamics of heavy metal accumulation (Cr, Cu, Zn, and Pb) in lake sediments closed to a cage farm located in Lake Ladoga, Russia. The results are useful for assessing the level of accumulation of metals in sediments under fish cages and their possible release into the water column. Furthermore, the findings are necessary for the development of methods for monitoring sediment pollution from fish farms and adjusting the fish feed diet in terms of microelement content.

MATERIALS AND METHODS

Study area

Lake Ladoga (60°50'34.0"N 31°27'35.0"E) spans an area of 17,680 km^2 with a water mass of 848 km^3, making it the largest freshwater body in Europe with a total catchment area of over 283,000 km^2. The northern part of the lake is highly suitable for trout cage culture, boasting more than 20 trout farms established within the last decade. The trout cage farm under study is situated in the skerry region of Yakkimvaar Bay within Lake Ladoga (Figure 1). Characteristics of both the farm and the bay were detailed in a previous publication (Lapenkov *et al*., 2023).

Sampling procedure

Fieldwork was conducted in winter (23–24 of February) and autumn (26–27 of October) 2021. These periods were chosen based on the results of previous research (Lapenkov *et al*., 2023) to show changes in metal contents in sediments in relation to intensity of fish feeding. During winter, as water temperature decrease, the amount of feed given to the fish was reduced, leading to a minimal thickness of the freshly accumulated organic matter layer in the sediments. In contrast, the feeding activity peaked in summer, leading to the organic layer reaching its maximum thickness by autumn.

The sampling scheme is presented in Figure 1. Four sites located directly adjacent to the cages (C1, C2, C3, C5) and three reference sites (R4, R7, R8) were studied. The cages of sites 1, 3 and 5 contain adult fish, site 2 housed juvenile fish. The distances from the farm to the reference sites were 150, 600, and 1,000 m to site S3, respectively.

The sediment cores were obtained using a gravity corer sampler. Immediately after collection, the sediment cores were segmented into 1–5 cm layers based on visual stratification analysis (Lapenkov *et al*., 2023). All sediment cores displayed a similar granulometric composition, characterized by gray argillaceous silt interspersed with a small amount of aleuritic particles. For preservation and subsequent analysis, the samples were placed in clean polyethylene bags and transported to the laboratory in a cool box.

The layers selected for total metal content analysis were determined based on the organic matter content (Lapenkov *et al*., 2023). Both the surface layer (with maximum organic matter content) and the deepest layers (background sediment with minimal organic matter content) were analyzed at each site. The percentage of the most labile forms of metals was measured in the surface layer.

Laboratory preparation and analysis of the sediments

The sediment samples were dried at room temperature for 48 h to analyze the labile form, and at 105 °C for 120 min to analyze the total content. After drying, the samples were sieved (2 mm) to remove gravel and large organic debris. Then each sample was ground in a jaspidean mortar to ensure homogeneity with passing a 0.1 mm sieve.

For the total content analysis, a mixture of strong acids ($HClO_4$, HF, HCl, and HNO_3) was used. The reagents and sample digestion times are outlined in a previous study (Guzeva *et al*., 2021). To ensure measurement quality, the certified standard material (sediments from Lake Baikal BIL-1–GSO 7126–94) was used (Petrov *et al*., 1999).

Labile (potential bioavailable) forms were extracted by ammonium acetate (1 M $CH_3CO_2NH_4$), known for extracting the most geochemically mobile forms of trace metals: exchangeable ions and specific sorbed metals (Guzeva *et al*., 2021). In this study, 0.5 g of air-dried sediment sample was mixed with ammonium acetate reagent at a 1:10 weight ratio and incubated for 24 hours at room temperature with stirring on a laboratory shaker. The decomposition of each sample was carried out in duplicate. The hygroscopic moisture content in the samples was determined to convert to absolutely dry weight.

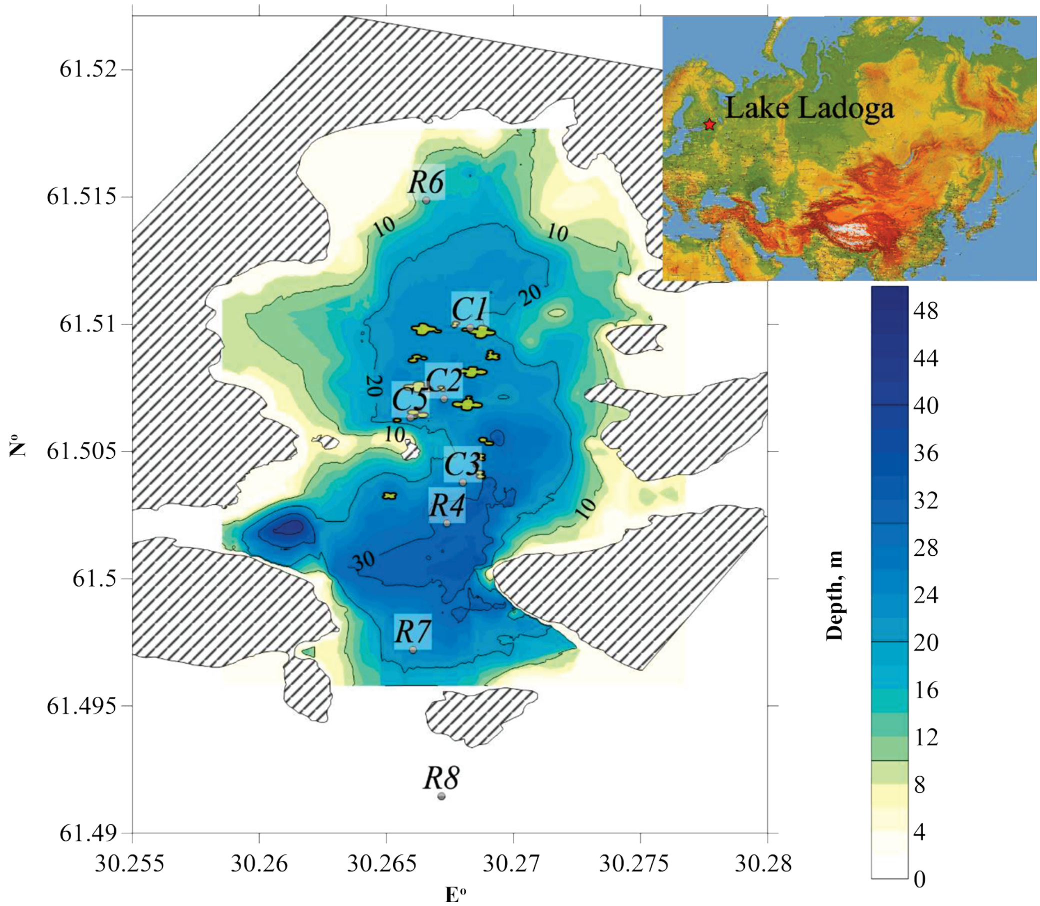

Figure 1. Location of the sampling sites, Lake Ladoga (Russia).

The content of trace elements in all extractions was measured by the mass spectral method using an XSeries 2 ICP-MS instrument by Thermo Ficher Scientifc (Waltham, MA, USA). The result was accepted as correct for the sample if the difference in values of duplicates did not exceed 20% (Slukovskii, 2020). Then, the mean value for each sample was calculated. The standard deviation (%) of the measurements of total content was as follows: Cr (0.3–2.5); Cu (0.9–2.7); Zn (0.2–1.9); Pb (0.4–2). The standard deviation (%) of the measurements of labile forms was as follow: Cr (0.5–5); Cu (0.4–4); Zn (0.3–3); Pb (0.6–6).

Assessment methods

The Pearson coefficient was calculated to assess the degree of correlation between organic matter content and the total content of the metals. The data on dispersed organic matter (DOM, mass. %) content in the layers of the sediment cores were taken from the previous research (Lapenkov *et al*., 2023).

The Mann-Whitney test is used to compare small samples ($n<30$). The test was used to reveal the differences in metal accumulation between cage and reference sites.

The geoaccumulation index (I_{geo}) was used to assess the accumulation level of the heavy metals in the sediments in the impact zone of the cage farm, as shown in Equation 1.

$$I_{geo} = \log_2[C/(1.5\times B)] \qquad (1)$$

where C is the concentration of metals in the studied sediment layer and B is the background concentration of the element, determined in the lowest layer of the sediment core (Müller, 1969). The level of metal pollution was estimated according to the scale: unpolluted ($I_{geo}\leq 0$), unpolluted to moderately polluted ($0<I_{geo}<1$), moderately polluted ($1<I_{geo}<2$), moderately to strongly polluted ($2<I_{geo}<3$), strongly polluted ($3<I_{geo}<4$), strongly to extremely polluted ($4<I_{geo}<5$), and extremely polluted ($I_{geo}>5$).

Sediment quality guidelines (SQGs) method was used to evaluate the total content of the heavy metals in terms of sediment toxicity for freshwater ecosystems (MacDonald *et al*., 2000; Li *et al*., 2015). SQGs include threshold effect concentration (TEC) and probable effect concentration (PEC). If the concentration of the metal is below the TEC, then dangerous effects rarely occur. If the content of the metal is higher than PEC, it suggests adverse effects are likely to occur frequently. The values of TEC/PEC are as follows: Cr–43.4/111; Cu–31.6/149; Zn–121/459; Pb–35.8/128.

The risk to the aquatic ecosystem, in terms of the mobility (bioavailability) of the metals was assessed using the indicator of the risk of secondary water pollution (RAC index) (Passos *et al*., 2011). This index takes into account the percentage of the most mobile forms of metals (exchangeable and specifically sorbed ions) extracted with ammonium acetate. Metals in these forms can be released into the water column when the ionic composition of the water changes or the pH decreases to acidic values. The scale consists of five levels of risk (percentage of labile forms from the total content of the metal):<1% –no risk, 1–10% –low risk, 10–30% –medium risk, 30–50% –high risk, and > 50% –very high risk.

RESULTS

Total content of the metals

The results of the total content analysis of the metals in the sediment cores are presented in Figures 2 and 3. The difference between the level of metal accumulation in February and October was not revealed at the reference sites R4, R7, and R8. Furthermore, Cr, Cu, Zn, and Pb are distributed quite homogeneously over the depth of the cores - the coefficient of variation does not exceed 30% for all elements (Table 1). The average value and the standard deviation were calculated for each metal at the reference sites.

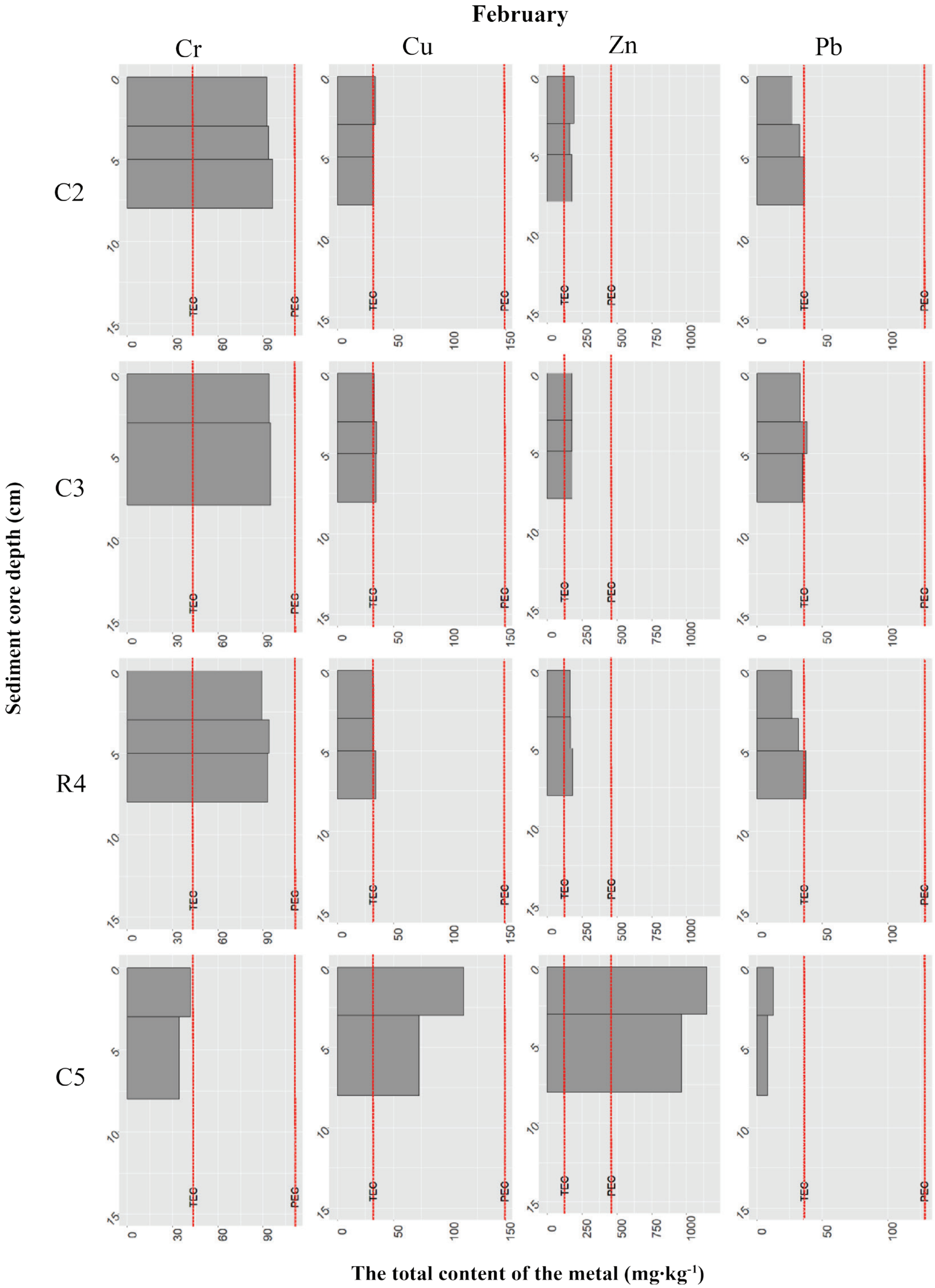

Figure 2. Total metal content in the sediment cores collected within the impact zone of a fish farm in February: C2, C3, C5 = cage sites; R4 = reference site. The red dotted line indicates TEC and PEC concentrations (Li *et al*., 2015).

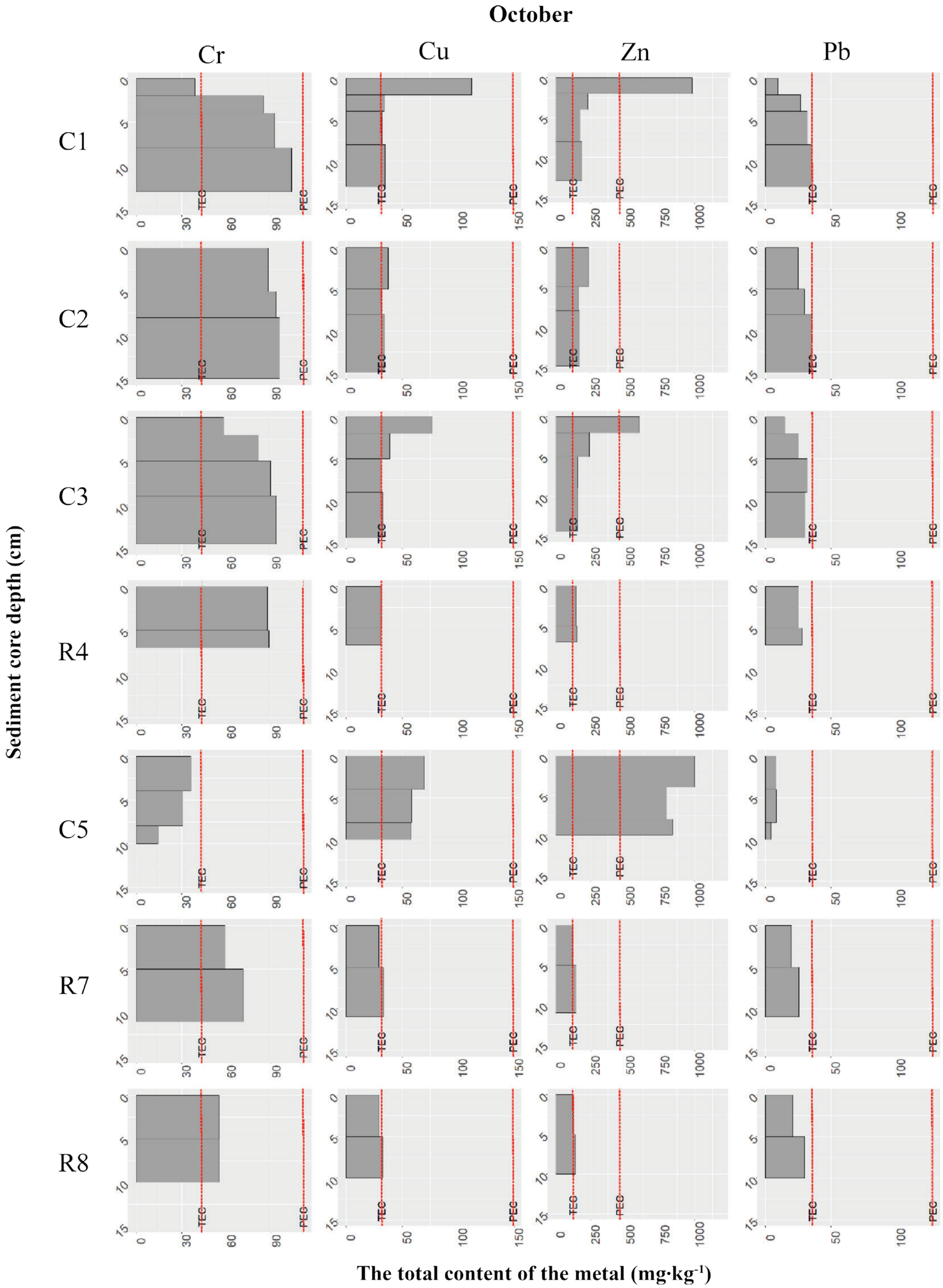

Figure 3. Total metal content in the sediment cores collected within the impact zone of a fish farm in October: C1, C2, C3, C5 = cage sites; R4, R7 and R8 = reference sites. Red dotted line indicates TEC and PEC concentrations (Li *et al*., 2015).

Table 1. Statistical parameters of the total content ($mg \cdot kg^{-1}$) of the metals in the sediments in the impact zone of cage farm (Lake Ladoga, Russia); n = number of samples for layer-by-layer analysis (Figure 2 and 3).

Samples	Parameters	Cr	Cu	Zn	Pb
Reference sites (n = 11)	Average (October + February)	78.2	31.8	150.6	27.8
	Standard deviation	16.3	1.6	18.5	5.5
	Coefficient of variation, %	21	5	12	20
Cage sites (n = 21)	Maximum concentration in October	103.3	112.3	998.8	35.4
	Maximum concentration in February	96.5	112.6	1148.6	38.2
	Average	75.7	43.3	331.2	25.5
Mann–Whitney test (*U*)*	Reference / Cage sites	97	**35**	**21**	106

Note: * = values less than critical are highlighted in bold

The seasonal dynamics of the accumulation of Zn and Cu in the top layers of sediment cores were noted at the cage sites C1, C2, C3, and C5. The most significant concentrations of Zn (more than 900 $mg \cdot kg^{-1}$) in the surface layers were observed at sites C1 and C5 in autumn. In general, the top 5 cm of all sediment cores taken in October are enriched in Zn by 2–10 times compared to the lower (background) layers and reference sites (Table 1, Figure 3). The exception is C5: a significant concentration of Zn was identified in all analyzed layers of the cores (more than 10 cm) in October and February (Figure 2 and 3). An increased content of Cu is also observed in the surface layers of sediment cores from cage sites (C1, C3, and C5). However, its concentration exceeds the reference values by no more than 2–3 times. The average values of the total content for Zn and Cu in cage sites significantly differ from reference sites. The calculation of the Mann-Whitney test is presented in the Table 1.

Seasonal and vertical dynamics in the accumulation of Cr and Pb were not revealed. Their contents in all layers of the cores taken at the cage sites are commensurate with the reference values (Table 1).

A significant positive correlation (n = 64; $\alpha = 0.01$) between organic matter (Lapenkov *et al.*, 2023) and Zn, Cu was found (Figure 4). Cr exhibited a significant negative correlation with DOM (mass. %), whereas a statistically significant correlation for Pb was not revealed.

I_{geo} index of the sediments was calculated for all sites (Table 2). The sediments of reference sites (R4, R7 and R8) were found to be unpolluted in all seasons ($I_{geo} \leq 0$) for all studied elements. For C5, the lower layers of the cores from the nearest C2 were considered as background values. No contamination of Cr and Pb was observed in the cage sites either in February or October. However, moderate to strong pollution levels ($1 < I_{geo} < 2$) were detected in the sediments at C5 for Zn in both February and October. Sediments were found to be unpolluted to moderately polluted ($0 < I_{geo} < 1$) for Cu at C5 in both February and October. Furthermore, the sediments at sites C1 and C3 were characterized by Zn (moderately polluted) and Cu (unpolluted to moderately polluted) enrichment only in autumn (October).

The average reference values for all elements (Table 1) are at the level of TEC (Li *et al.*, 2015). An excess of PEC values for Zn was observed in October at sites C1 and C3, and both in October and February at C5 (Figure 2 and 3).

Labile forms of the metals

The concentration of labile forms (% of the total content) of the studied metals in the surface sediment layers is presented in Figure 5. The largest percentage of labile forms was found for Zn. It should be noted that its relative mobility at the reference sites is commensurate with the mobility at cage sites. However, the absolute content (mg·kg^{-1}) of labile forms in the cage sites was 3–6 times higher than in the reference ones. In general, the RAC index exceeded the moderate risk in only 2 samples. The mobility of other elements was low at all sites except site 4, where the RAC index of Cu was more than 20%.

Table 2. I_{geo} values for heavy metals in sediments of cage (C1, C2, C3, C5) and reference (R4, R7 and R8) sites.

Month	Sites	Cr	Cu	Zn	Pb
February	C2	-0.6	-0.5	-0.5	-1.0
	C3	-0.6	-0.7	-0.6	-0.7
	C5	-1.8	1.2	2.1	-2.1
	R4	-0.6	-0.7	-0.7	-1.1
October	C1	-2.0	1.1	1.8	-2.5
	C2	-0.7	-0.4	-0.1	-1.1
	C3	-1.3	0.7	1.4	-1.7
	C5	-2.0	0.5	2.0	-2.7
	R4	-0.6	-0.7	-0.7	-1.0
	R7	-0.9	-0.8	-0.8	-1.0
	R8	-0.6	-0.7	-0.8	-1.1

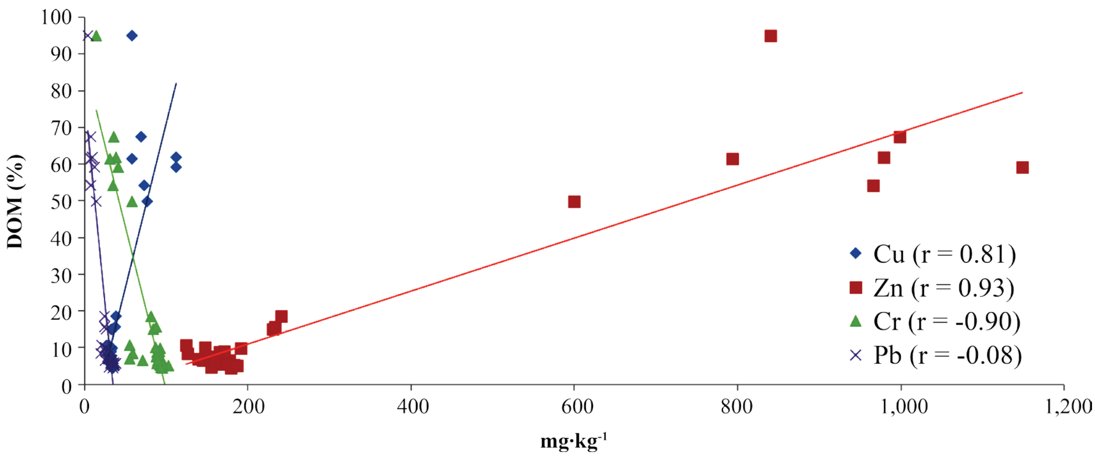

Figure 4. The Pearson correlation (r) between Zn, Cu and organic matter content (DOM, mass. %). The values of DOM were taken from the previous research (Lapenkov *et al.*, 2023).

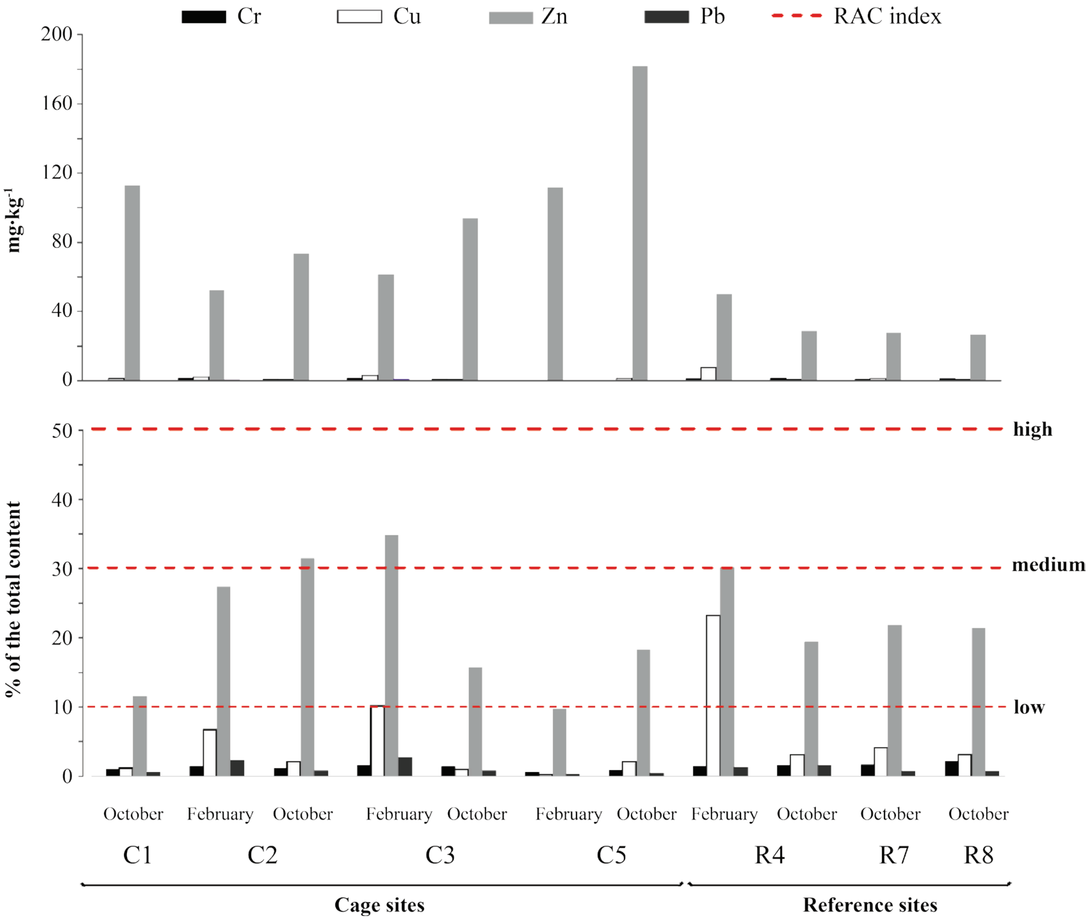

Figure 5. The content (absolute and percentage) of labile forms of the metals in the surface layer of the sediment cores.

DISCUSSION

The study found a significant positive correlation between Zn and Cu with organic matter among the examined metals. This suggests that these elements mainly enter the sediments under the farm cages with organic waste. Previous research has shown seasonal dynamics in the quantity of organic material that enters the sediments under the cages (Lapenkov *et al*., 2023). This pattern also reflects in the seasonal variations in the intensity of Zn and Cu accumulation within these sediments.

According to the I_{geo} index, Zn and Cu are pollutants in the sediments under the cages. Similar research on cage farms also demonstrated a correlation between organic matter and Zn, Cu in surface sediments (Melo Júnior *et al*., 2023). The excessive input of these elements into sediments might be attributed to their high concentration in trout feed and poor digestibility by fish. For instance, the metabolic absorption rate of tilapia cultured in net cages of Cas-tanhão weir (Brazil) was reported to be 2.61% for Zn and 47.4% for Cu (Oliveira *et al*., 2017). The metabolic requirements of Rainbow trout necessitate specific levels of essential minerals in their diet, with approximately 3 mg·kg^{-1} of Cu, 12–13 mg·kg^{-1} of Mn, 0.15–0.38 mg·kg^{-1} of Se, and 15–30 mg·kg^{-1} of Zn (Lall and Kaushik, 2021). Therefore, any surplus or unutilized portions of these chemical elements are subsequently discharged into the surrounding environment.

Zinc was found to accumulate significantly in the upper 4–5 cm of sediments under the cages, especially towards the end of October when the intensive feeding season ended. During this period, the potential toxicity of the surface sediment layers increases significantly due to Zn excess, with Zn concentrations in the top layers of the cores from the cage sites being 4–10 times higher compared to the lower (background) layers.

Elevated concentrations of Zn and Cu in sediments beneath fish farms have been observed in other studies as well. In the Changshou Reservoir (Chongqing, China), during the winter period, the surface layer of bottom sediments (0–10 cm) showed maximum Zn and Cu contents exceeding background levels by 2.5 to 3 times (Xie *et al.*, 2020). On the other hand, in the Keban Reservoir (Turkey), sediments collected directly under fish farms, even in late August (after three months of intensive fish feeding), exhibited low Zn and Cu concentrations that did not surpass background levels (Varol, 2019). This discrepancy could potentially be explained by differences in feed composition and the better flow regime of the water body.

Chromium is also present in fish feed but its correlation with organic matter was negative. In autumn, the Cr content was at the level of reference concentration, even in the fresh-organic layers of the sediment under the cages (Table 1). Therefore, the content of Cr in farm waste is very low. The research also showed that Cr has a positive correlation with organic matter; however, the increase in its concentrations compared to the control area was significantly less than that of Zn and Cu. This can be attributed to the low content of chromium in fish feed and its relatively high metabolic absorption rate in fish.

Lead did not show significant accumulation in sediments under the cages and did not pollute the sediments in the studied area of the fish farm. In general, Pb concentrations in sediments were comparable to other studies on freshwater cage farms (Xie *et al.*, 2020; Melo Júnior *et al.*, 2023).

The farm's spatial influence on heavy metal pollution is limited to the studied bay. The accumulation of excess metals and organic matter was observed directly under the cages, with the oldest cages at C5 (continuously present since around 2010) showing the highest enrichment in Zn and Cu. Relatively cleaner sediments under the cages were found at C2, where juvenile fish are kept. At 150 m from the cages (site R4), metal concentrations were comparable to more distant reference sites R7 and R8. Furthermore, statistical calculation (Table 1) reveals that in the cage sites, the average concentrations of Zn and Cu are higher than in the reference sites.

Seasonal dynamics of metal accumulation in the sediments were observed only for Zn and Cu in the cage sites. In October, the highest concentrations of the metals were primarily found in the upper 3–5 cm of the cores. However, by February, as fresh sediments gradually compacted, metal concentrations notably decreased, and reached the reference level. The exception was site C5, where the layer of contaminated sediments was the thickest (over 10 cm). Elevated concentrations of Zn (exceeding PEC) and Cu (2–3 times higher than the background levels) were observed there even in February, when the feeding intensity is minimal. The cages at this site are the oldest on the fish farm and presumably have not been moved from this location for more than 5 years. Thus, a layer of metal-rich sediments has already accumulated underneath them and exists there all year round. It is necessary to consider that high concentration of Zn poses a risk to benthic biota beneath the cages.

The content of labile forms of Cu, Cr, and Pb was no more than 25% of the total content of the metals at all studied sites. The labile forms of Zn exhibited a higher content, but it did not exceed 33%. However, according to the RAC index, the risk of secondary pollution of the water column is no more than medium level even for Zn. The proportion (%) of labile forms of the elements in the total content in the cage sites is comparable to the reference area (Figure 5). However, the absolute content ($mg\cdot kg^{-1}$) of labile Zn is much higher under the cage than at reference sites, especially at C5. It should be noted that strongly reducing conditions (Eh values were from -100 to -250 mV) were formed in the surface sediments during winter (Lapenkov *et al*., 2023). The anoxic environment contributes to the geochemical mobility of Zn and Cu. Thus, if the high input of metals with farm waste continues, there is an increased risk of migration of potentially toxic elements into the pore and bottom water from highly polluted sediments.

CONCLUSION

This study provides valuable insights into the seasonal dynamics of heavy metal (Cr, Cu, Zn, and Pb) accumulation in the sediments of a lake trout farm's impact zone. The research demonstrates significant pollution of the surface sediments under the cages with Zn and Cu, which are present in fish feed and enter the environment through organic waste from the fish farm. According to the I_{geo} values, surface layers of sediments under some cages were characterized by strong levels of Zn contamination and medium levels of Cu. Notably, Zn accumulates significantly in surface sediments towards the end of the active feeding period, resulting in an increase in potential sediment toxicity. Consequently, Zn and Cu can be utilized as geochemical markers to assess the thickness of the accumulated "contaminated" sediment layer in the cage farm's impact zone. The results indicate that there is no pollution of sediments with Cr and Pb in the studied area.

The percentage content of labile forms of Cu, Cr, Pb, and Zn was no more than 33% at all studied sites. However, the absolute content ($mg\cdot kg^{-1}$) of labile Zn is much higher under the cage than at reference sites.

The spatial influence of heavy metal pollution from the fish farm is local, being confined to the studied bay of Ladoga Lake. This comprehensive layer-by-layer analysis contributes to our understanding of the environmental impact of fish farms and highlights the importance of monitoring heavy metal accumulation in sediments. The findings underscore the need for sustainable practices and effective waste management in aquaculture to mitigate potential adverse effects on the environment.

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