Зборник Матице српске за природне науке / Matica Srpska J. Nat. Sci. № 139, 73—87, 2020

UDC 546.49(282.243.742 Tisa)
https://doi.org/10.2298/ZMSPN2039073S

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MERCURY POLLUTION OF SEDIMENTS FROM THE RIVER TISA (SERBIA)

ABSTRACT: Mercury (Hg) has been listed as a global high priority pollutant by many international organizations due to its mobility and persistence in the environments and high toxicity to organisms. This research was conducted with the aims to determine: (i) total Hg content (THg) and its spatial distribution in sediments of river Tisa along the river course, (ii) possible sources of THg and (iii) degree of THg pollution in sediments from the river Tisa through different criteria. Total Hg in the sediments ranged from 0.07 to 0.49 mg kg^{-1}, with mean ± S.D. value of 0.26 ± 0.10 mg kg^{-1}. The highest mean value of THg (0.30 mg kg^{-1}) was found in the lower stream, while the lowest (0.13 mg kg^{-1}) was found in the tributary. According to Principal Component Analyses (PCA) strong positive loading of metals in all parts of the river Tisa is mainly controlled from the same sources. These sources are related to anthropogenic activities based on calculated Enrichment Factor (EF) values. Total Hg are higher than background value. According to the Republic of Serbia official standard, THg values of river Tisa sediments were within the range of maximum permissible values. Compared with National Oceanic and Atmospheric Administration (NOAA) guideline, 80.49% of sediment samples indicated that THg in the river Tisa sediments represented minimal and possible risk towards the living organisms. Integrating the results of pollution assessment, it could be concluded that THg in river Tisa sediments in Serbia demonstrates considerable

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contamination according to Geoaccumulation Index (Igeo), and Contaminant Factor (CF), and high pollution risk according to Potential Environmental Risk Index (PERI).
KEYWORDS: mercury, risk assessment, sediments, river Tisa

INTRODUCTION

Mercury (Hg) has been listed as a high priority pollutant by many international organizations due to its persistence in the environments and high toxicity to organisms (Jiang et al., 2006). A global effort to common start in solving this problem has been finalized by the adoption of the Minamata Convention on Mercury (UNEP, 2019). This Convention requires that countries around the world control both new and existing sources of Hg emission and monitor the effectiveness of those controls. The control and management of Hg pollution require both global and regional efforts. Ninkov et al. (2017) studied total Hg content (THg) in agricultural soils from the aspect of soil suitability for the production of healthy and safe food, and its spatial distribution in different parts of Vojvodina province (Serbia). The same authors found the highest THg in alluvial plains of the Danube, Sava and Tisa. In aquatic systems, sediment is usually considered as an ultimate sink of pollutants discharged from land-based sources, such as heavy metals and persistent organic pollutants. Mercury can accumulate in sediments and be released to the surrounding media posing a risk on the living world. In rocks and sedimentary deposits, Hg is characterized by very low natural background concentrations. Thus, an anthropogenic impact can easily be detected by a significant increase of its concentrations. Based on previous research focused on the analysis of background levels of heavy metals in the river Tisa sediments (Štrbac et al., 2018), and in the view of the importance of regional studies of THg, investigation of THg in the river Tisa sediments in this study continued with other aspects of observation. In the present study, a great number of samples were observed, and in addition, a direct analytical method with a lower detection threshold was applied (Nedić et al., 2019). This research was conducted with the aims to determine: (i) THg and its spatial distribution in sediments of river Tisa along the river course, (ii) possible sources of THg and (iii) degree of THg pollution in sediments from the river Tisa through different criteria.

MATERIALS AND METHODS

Study area and sample collection

A total of 41 sediment samples were collected from twenty sites. Thirty-seven samples were taken from river Tisa (13 from upper and 12 each from middle and lower stream). Sampling transect was about 150 km long and stream deposits were collected approximately every 10 km (Figure 1). Four sediment samples were taken from tributaries Begej and Jegrička River and crossed

meander Mrtva Tisa. The sediment samples were taken from depth 0–60 cm depending on the site. The sediment samples were sectioned at 15 cm intervals, taking one to four samples per site. After sampling, the samples were stored in polyethylene bags. The initial quantity of samples was approximately 1.5 kg. The samples were air dried in the laboratory, ground in a mortar and passed through a 0.063 mm mesh in order to achieve consistent physical properties. Sediment samples were collected from September to November 2010. A global positioning system (GPS, Garmin Etrex summit HC, Kansas City, USA) was used to target the different sites.

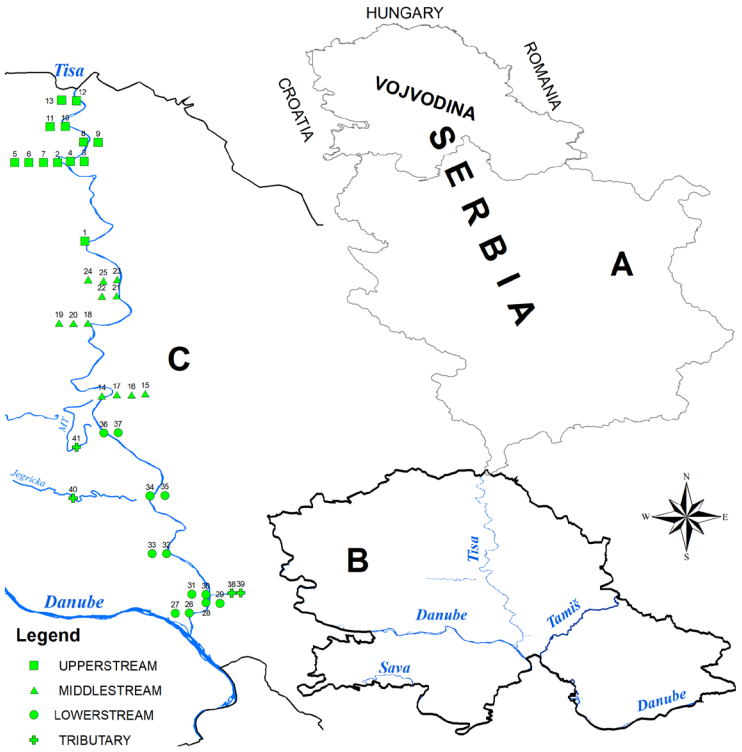


Figure 1. A – Study site; B – Flow of the river Tisa in the Vojvodina province; C – Sites of sediment samples collection.

Laboratory analysis

Particle size distribution was determined in <0.063 mm fraction by the pipette method (Obradović and Vasić 1988). Back-titration with 0.5 N NaOH and phenolphthalein as an indicator was used to determine carbonate content. For THg the samples were analyzed using Direct Mercury Analyzer DMA 80 Milestone as described in Nedić et al. (2019). Three replicates of each sample were used. Quality assurance and quality control (QA/QC) were conducted by

certified reference material BCR 142R (light sandy soil) – EC, JRC, Institute for Reference Materials and Measures. Analysis of BCR samples achieved analytical precision, measured as relative standard deviation, of 0.0025%. The accuracy was within interval 92.84–109.70%, and recovery was 101.11%. The limit of detection (LOD) in our study was 0.0033 mg Hg kg^{-1}. Provided QA/QC procedure verified the validity of applied analytical method.

Assessment of THg contamination in sediment

Background value (Štrbac et al., 2018), national legislation (*Official Gazette of RS* no. 50, 2012), National Oceanic and Atmospheric Administration (NOAA) guideline (Bolaños-Alvarez et al., 2016), and calculation of Enrichment Factor (EF), Geoaccumulation Index (Igeo), Contaminant Factor (CF), and Potential Environmental Risk Index (PERI) were used for the assessment of THg contamination in river Tisa sediments.

According to Ergin et al. (1991) EF is calculated in the following way:

$$EF = (C_{THg}/C_{Al})_{samples} / (C_{THg}/C_{Al})_{background\ area}$$

where $(C_{THg}/C_{Al})_{samples}$ is the ratio of THg (C_{THg}) to the Al concentration (C_{Al})$_{samples}$ in the sediment sample and $(C_{THg}/C_{Al})_{background}$ value is the same ratio in an unpolluted reference sample. The EF classes are: <1 no enrichment; 1–3 minor enrichment; 3–5 moderate enrichment; 5–10 moderately severe enrichment; 10–25 severe enrichment; 25–50 very severe enrichment; >50 extremely severe enrichment (Birch and Olmos, 2008).

The Igeo was used to assess Hg contamination in sediments, and is expressed by Müller (1969) as follows:

$$Igeo = \log_2 C_{THg}/1.5 \times B_n$$

where C_{THg} is the measured THg in the sediment, B_n is the THg geochemical background value of each basin. The constant 1.5 allows us to analyze natural fluctuations in the content of a given substance in the environment and to detect very small anthropogenic influences. Igeo classes are: <0 unpolluted; 0–1 unpolluted to moderately polluted; 1–2 moderately polluted; 2–3 moderately to highly polluted; 3–4 highly polluted; 4–5 highly to very highly polluted; 5–6 very highly polluted (Yaqin et al., 2008).

The PERI was proposed by Hakanson (1980). It has been applied to evaluate the harm of heavy metals in the sediments. The method was described as follows:

1. Contamination Factor (CF) is the ratio obtained by dividing the concentration of metal (C) in the sediment by the background value (Co) (Hakanson, 1980):

$$CF = C / Co$$

According to Hakanson (1980), CF <1 indicates low contamination; 1< CF <3 is moderate contamination, 3< CF <6 is considerable contamination, and CF >6 is very high contamination.

2. The Potential Ecological Risk Index for the single heavy metal pollution (E_i) was described as follows:

$$E_i = T \times CF$$

where T is the toxic-response factor, and is 40 for Hg (Hakanson, 1980), and CF is contamination factor. E_i classes suggested by Håkanson (1980) were: <40 low risk; 40≤ E_i <80 moderate risk; 80≤ E_i <160 considerable risk; 160≤ E_i <320 high risk; ≥320 very high risk.

Statistical analysis

All statistical parameters of descriptive statistics were calculated. The significance of differences in measured parameters between four different part of the river Tisa (upper, middle, lower stream and tributary) was determined using Fisher's LSD test ($p \leq 0.05$). Multivariate analysis performed on the data include Principal Component Analyses (PCA). PCA was used to characterize the concentration of THg in relation to grain size and organic matter (OM) and $CaCO_3$ in the upper, middle and lower streams of the river, as well as the main tributaries and to assess possible contribution sources of THg as well as other heavy metals in sediments of the river Tisa. PCA with varimax normalized rotation was carried out separately for the four different parts of the river Tisa: upper, middle, downstream and tributary to identify the factors influencing each of them. Only factors with an Eigen value greater than 1 were considered significant. Variables with a factor loading greater than 0.7 were interpreted as being meaningfully correlated with the factor. A scatter plot of PCA mean score values was used to graphically display the positions of investigated heavy metals in environmental space. For statistical analysis Statistica version 12 (Dell Inc. 2016) was used.

RESULTS AND DISCUSSION

THg distribution in sediments of the river Tisa along the river course

Total Hg in the river Tisa sediments is shown in Figure 2. Results for the minimum, maximum, mean, and median values, standard deviation, and coefficient of variation of the THg are shown in Table 1.

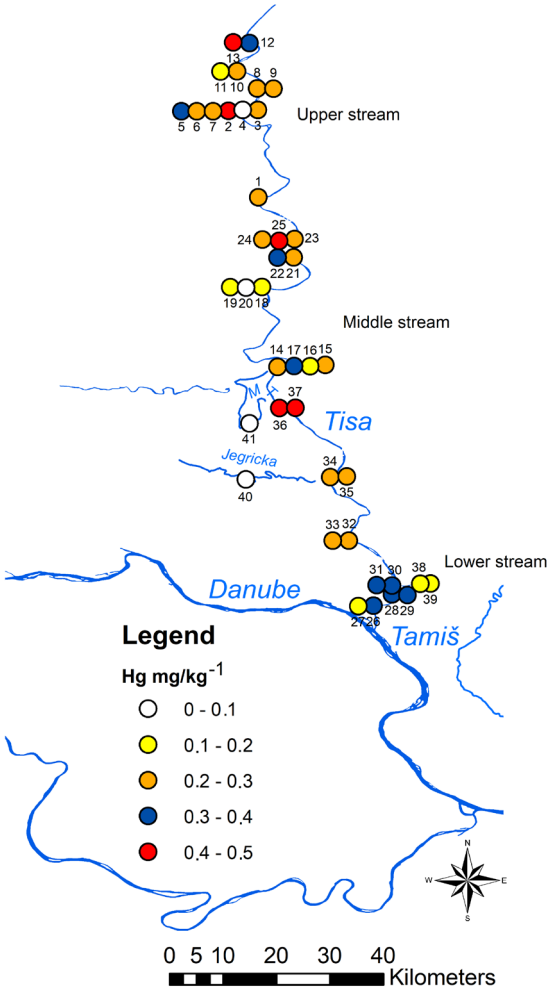


Figure 2. Total Hg (mg kg^{-1}) in the river Tisa sediments along the watercourse

Table 1. Statistical summary of THg concentrations (mg kg^{-1}) in the river Tisa sediment samples

Statistical parameters	Value
Number of samples	41
Minimum value	0.07
Maximum value	0.49
Mean value	0.26
Median value	0.27
Standard deviation (S.D.)	0.10
Coefficient of variation (CV) [%]	39.42

Total Hg in the sediments ranged from 0.07 to 0.49 mg kg^{-1}, with mean ± S.D. values of 0.26 ± 0.10 mg kg^{-1} and median value of 0.27 mg kg^{-1} (Table 1). The lowest concentration (0.07 mg kg^{-1}) was found in the sample 20 in the middle stream, while the highest concentration (0.49 mg kg^{-1}) was found in the sample 37 in the lower stream of the river (Figure 2). The highest mean value of THg (0.30 mg kg^{-1}) was found in the lower stream, while the lowest concentration (0.13 mg kg^{-1}) was found in the tributary. The mean value was obtained based on the measured THg values in the upper, middle and lower stream of the river. Figure 1 shows the distribution of the samples depending on the river flow. Based on THg along the river Tisa streams, tributaries have the lowest content with statistical difference compared to other parts of the river stream. Moreover, the tributaries have the highest percentage of clay and carbonate content (Table 2). Gu et al. (1998) emphasizes that Hg pristine concentrations in igneous and sediment or soil samples vary between 0.08 and 0.4 mg kg^{-1}. According to Ullrich et al. (2001) THg in surface sediments of uncontaminated or less contaminated rivers range from 0.02 to 0.4 mg kg^{-1}, and can be as high as 100 mg kg^{-1} in urban, industrial or mining areas. Total Hg concentrations obtained in this study are lower than Hg contents reported from the river Sava (0.2–0.6 mg kg^{-1}) (Milačič et al., 2010), and the Danube (<0.10–2.37 mg kg^{-1}) (Woitke et al., 2003), and are similar to Hg contents reported from Serbian rivers and artificial lakes (<0.0001–0.72 mg kg^{-1}) (Sakan et al., 2017), and the river Tisa (0.14–0.36 mg kg^{-1}) (Štrbac et al., 2018).

To characterize the THg in relation to grain size, organic matter (OM) and $CaCO_3$ in the upper, middle and lower stream of the river, as well as in the main tributaries PCA was used. PCA defined two groups, which explained 74.22% of the total variation (Figure 3).

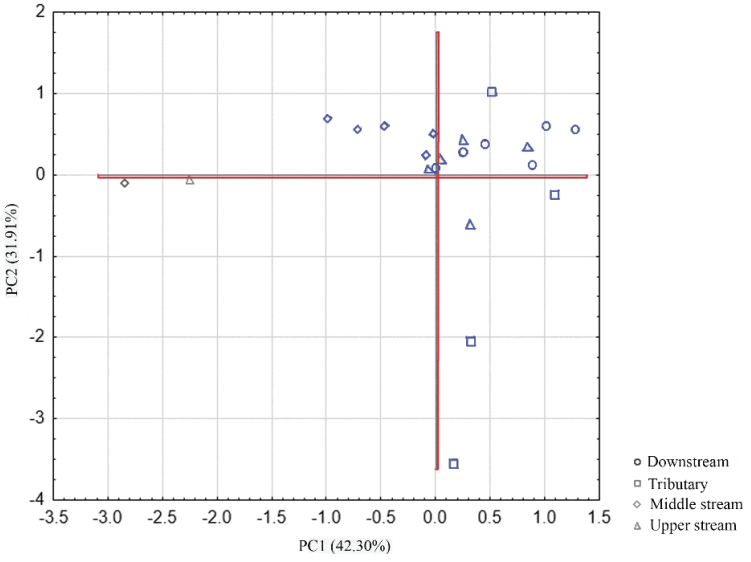


Figure 3. The projection of the cases of the first two components of the PCA

The first principal component explained 42.30% of the variation. It was defined by the concentration of sand, silt and clay. The projection of the cases for the first two components showed that lower stream and tributary could be clearly separated from upper and middle stream (Figure 3). Based on comparison of lower stream and middle stream, in lower stream the significantly higher concentration of silt was found, while in middle stream a significantly higher concentration of sand was found (Table 2).

Table 2. Sediment characteristics of the river Tisa (mean value ± standard error)

	Lower stream	Middle stream	Upper stream	Tributary
THg [mg kg^{-1}]	0.30±0.04 **a**	0.26±0.05 **a**	0.20±0.03 **a**	0.13±0.02 **b**
Sand [%]	14.24±2.96 **b**	44.69±1.83 **a**	27.25±9.29 **ab**	14.31±3.66 **b**
Silt [%]	60.73±1.91 **a**	39.01±0.62 **b**	53.19±1.77 **ab**	52.93±2.44 **ab**
Clay [%]	25.03±2.23 **ab**	16.29±2.02 **b**	19.55±3.15 **b**	32.76±5.62 **a**
$CaCO_3$ [%]	3.59±0.10 **b**	3.78±0.24 **b**	5.13±0.64 **b**	9.12±3.38 **a**
Organic matter (OM) [%]	0.20±0.004 **ns**	0.18±0.006 **ns**	0.19±0.001 **ns**	0.22±0.029 **ns**

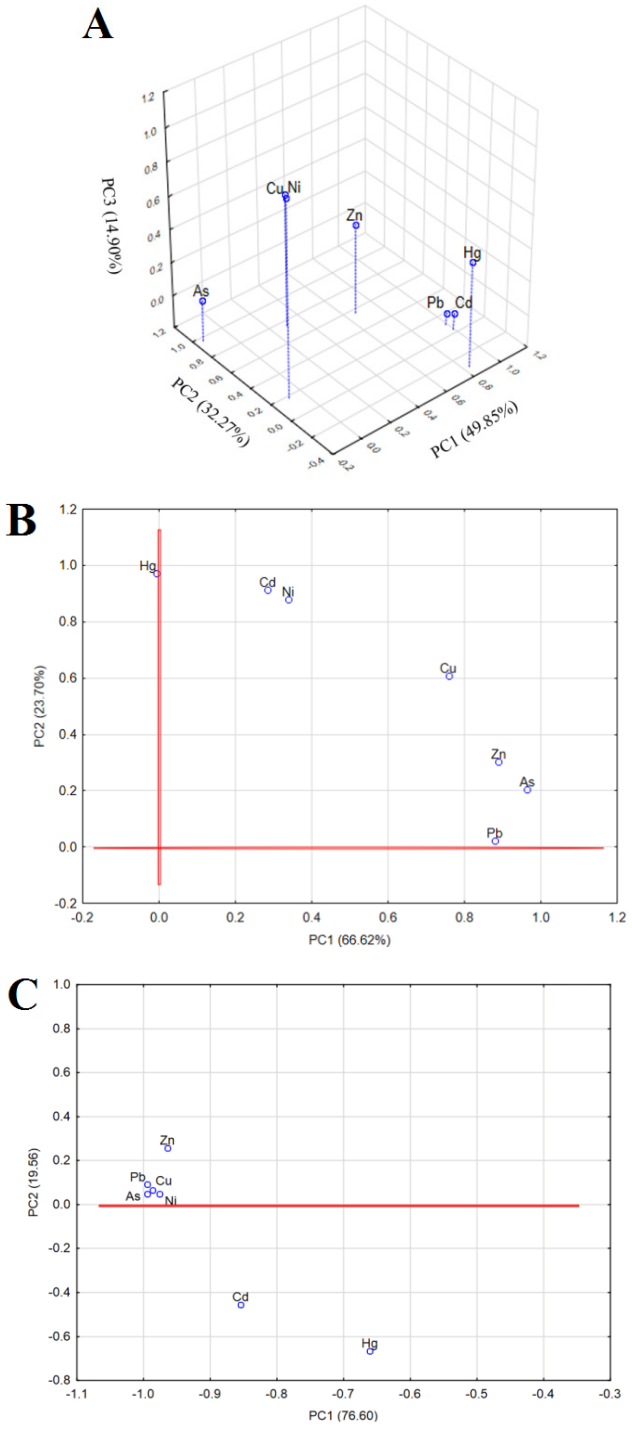
* Different letter indicates that differences between different parts of the river Tisa are significant according to Fisher's LSD test ($p \leq 0.05$), ns – not significant

The second principal component explained 31.91% (Figure 3) of variation due to the variability in $CaCO_3$ and OM and showed clear separation of the middle stream from other investigated parts of the river Tisa. Tributaries have the highest concentrations of clay, $CaCO_3$ and OM. Fisher's test showed no significant differences in OM between different parts of the river Tisa (Table 2). In the river Tisa sediments Hg is likely to be transported primarily by the small particles. Tansel and Rafiuddin (2016) found that heavy metals concentrations were directly correlated with particle size. Fine sediments (<0.106 mm) can accumulate more than 10 times the levels of Hg in comparison to the sediments that are greater than 0.850 mm. The anthropogenic activities in the area of the samples with minimum and maximum values of THg concentration (samples 20 and 37) do not present other likely sources of Hg pollution, variation coefficient CV (39.42 %) points out small heterogeneity of tested sediment samples (Table 1), so the observed increase could be explained by change in the grain size of surface sediments. In the sample 20 percent of clay was 6.67%, and in the sample 37 it was 32.50%. Fine-grained sediments have a higher specific surface area of clay particles than coarser sediments, which can increase the ability of metals to associate (Liu et al., 2017). However, the lowest mean THg was found in tributaries, where the highest percentage of clay was present (Table 2). In addition to the highest percentage of clay, the highest percentage of $CaCO_3$ was also recorded. For remediating pollution of heavy

metals, calcium carbonate-enriched materials (e.g., mussel shells) have recently been introduced (Wang et al., 2019). Peña-Rodríguez (2010) have published the study that high levels of Hg could be removed using calcined mussel shell, although this material contained 7% of aragonite even after calcination (Peña--Rodríguez et al., 2013).

Possible sources of THg content

Total Hg obtained in the upper, middle and lower streams of the river, as well as the main tributaries, were compared with content of As, Cd, Cu, Ni, Pb, and Zn using PCA to establish possible sources of Hg. The contents of As, Cd, Cu, Ni, Pb, Zn in sediments were investigated in samples collected from the same sites during the same year and previously were reported in Štrbac et al. (2018). PCA of the fourth datasets defined three PCs for the lower stream and two PCs for the middle stream, upper stream and tributary which contributed to 97.03%, 90.31%, 96.15% and 98.26% of the total variance. For the dataset referring to lower stream group, the first principal component explained 49.85% of the total variance (Fig. 4A) and has strong positive loadings on Hg, Cd and Pb. The second principal component accounted for 32.27% of variation due to the variability in the concentrations of As, Cu, and Ni. The third principal component explained 14.90% of variation and is formed by Zn. For the second group (middle stream) PC1 (66.62% of total variance) is dominated by As, Cu, Pb and Zn. PC2 has strong positive loadings on Hg, Cd, and Ni explaining 23.70% of total variance (Fig. 4B). PC1 of the upper stream has strong positive loadings on As, Cd, Cu, Ni, Pb and Zn explaining 76.60% of the total variance. The second PC (19.56% of total variance) was related only to the Hg (Figure 4C). In fourth group – tributary, PC1 is dominated by Hg, Cu, Ni, Pb and Zn accounting for 74.68% of the total variance, while PC2 explaining 23.58% due to As (Figure 4D). According to PCA strong positive loading of examined metals in all parts in the river Tisa are mainly controlled from the same sources.


A
PC3 (14.90%)
PC2 (32.27%)
PC1 (49.85%)
Cu
Ni
Zn
Hg
As
Pb
Cd
B
PC2 (23.70%)
PC1 (66.62%)
Hg
Cd
Ni
Cu
Zn
As
Pb
C
PC2 (19.56)
PC1 (76.60)
Zn
Pb
Cu
As
Ni
Cd
Hg

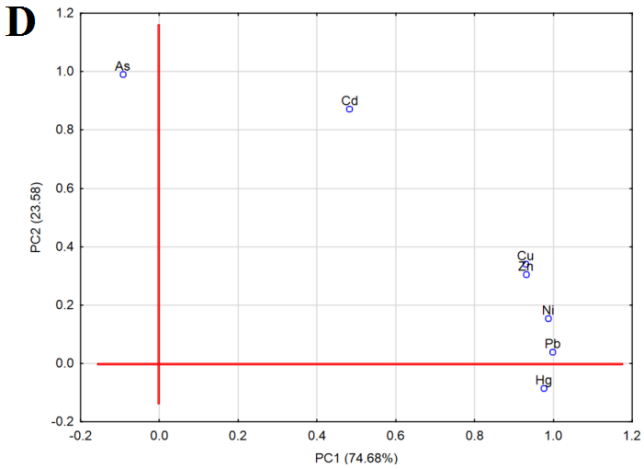


Figure 4. PCA loadings and score plots of metals for groups: A – lower stream; B – middle stream; C – upper stream; D – tributaries.

Degree of THg pollution in sediments from the river Tisa through different criteria

To establish the intensity of anthropogenic influence on the river Tisa sediments, the results of this study were compared with the background value, national legislation, and NOAA guideline. Total Hg were obviously higher than mean background value (0.065 mg kg^{-1}) (Štrbac et al., 2018). According to the official standard on the limit levels of pollutants in the Republic of Serbia, THg in the river Tisa was within the range of maximum permissible values (*Off. Gazette of RS* no. 50, 2012). Maximum permissible value is the concentration of an individual pollutant or group of pollutants above which negative environmental impacts are expected (*Off. Gazette of RS*, no. 50, 2012). Compared with NOAA guideline, 80.49% (Table 3) of sediment samples showed THg between the Effects Range-Low (ERL) and Effects Range-Median (ERM) values, indicating that THg in the river Tisa sediments represented minimal and possible risk towards organisms. The NOAA guidelines classified the sediment: rarely (<ERL), occasionally (≥ERL and <ERM) or frequently associated with adverse biological effects (>ERM) in relation to the concentrations of pollutants. Concentrations lower than ERL represent a minimal effect; those between ERL and ERM represent a possible effect; and those above the ERM represent a probable effect. The ERL and ERM toxicity values for THg as reported by NOAA are 0.170 mg kg^{-1} and 0.486 mg kg^{-1}, respectively (Bolaños-Alvarez et al., 2016). Degree of THg contamination was also assessed by pollution indices EF, Igeo, CF and PERI. EF of THg ranged from 1.06 to 6.75. EF results showed that 31.71% of samples showed minor enrichment, 53.66% moderate enrichment, and 14.63% severe enrichment (Table 3). However, an EF >1.5 indicates that a significant portion of the heavy metals was delivered

Table 3. Degree of THg pollution in sediments from the river Tisa.

NOAA guidelines (Bolaños-Alvarez et al., 2016)							
	<ERL		≥ERL and <ERM			>ERM	
Percentage of examined sediment samples	19.51%		80.49%			–	
EF (Birch and Olmos, 2008)							
	no enrichment	minor enrichment	moderate enrichment	moderately severe enrichment	severe enrichment	very severe enrichment	extremely severe enrichment
Percentage of examined sediment samples	–	31.71%	53.66%	14.63%	–	–	–
Igeo (Yaqin et al., 2008)							
	unpolluted	unpolluted to moderately polluted	moderately polluted	moderately to highly polluted	highly polluted	highly to very highly polluted	very highly polluted
Percentage of examined sediment samples	9.76%	39.02%	51.22%	–	–	–	–
CF (Håkanson, 1980)							
	low contamination		moderate contamination		considerable contamination		very high contamination
Percentage of examined sediment samples	–		34.15%		53.66%		12.19%
Ei (Håkanson, 1980)							
	low risk		moderate risk	considerable risk	high risk		very high risk
Percentage of examined sediment samples	–		12.19%	34.15%	53.66%		–

from non-crustal materials, thus these heavy metals were delivered by other sources, like point and non-point pollution (Barbieri, 2016). In this sense, THg in the river Tisa sediments has enrichment phenomenon with respect to the background value and mainly originates from anthropological sources. The Igeo index for the sampling sites ranged from -0.31 to 1.61. Igeo results showed that 51.22% of all sediment samples were moderately polluted, 39.02% unpolluted to moderately polluted, and 9.76% unpolluted (Table 3). According to the category of CF, 12.19% samples were at a very high contamination level, 53.66% samples were considerably contaminated, while 34.15% were moderately contaminated (Table 3). Specifically, 53.66% of sediment samples were within high pollution risk, 34.15% in considerable pollution risk, and 12.19% in moderate risk according to PERI (Table 3).

CONCLUSION

Total Hg concentrations in the sediments ranged from 0.07 to 0.49 mg kg^{-1}, with mean ± S.D. value of 0.26 ± 0.10 mg kg^{-1}. The highest mean value of THg (0.30 mg kg^{-1}) was found in the lower stream, while the lowest (0.13 mg kg^{-1}) was found in the tributary. In the river Tisa sediments THg is likely to be transported primarily by the small particles. The anthropogenic activities in the area of the samples with minimum and maximum values of THg do not present other likely sources of Hg pollution of the river Tisa, so the observed increase could be explained by change in the grain size of surface sediments in this area. According to PCA, strong positive loading of metals in all parts in the river Tisa is mainly controlled from the same sources. These sources are related to anthropogenic activities based on calculated EF values. EF indicates that THg concentrations in the river Tisa sediment have enrichment phenomenon. Total Hg are higher than mean background value. According to the Republic of Serbia official standard THg in the river Tisa was within the range of maximum permissible values. Compared with NOAA guideline, 80.49% of sediment samples indicated that THg in the river Tisa sediments represented minimal and possible risk towards the living organisms. Integrating the results of pollution assessment, it could be concluded that THg in river Tisa sediments in Serbia show considerable contamination according to Igeo and CF, and high pollution risk according to PERI.

ACKNOWLEDGEMENT

This work was financially supported by the Ministry of Education, Science and Technological Development of the Republic of Serbia (Grant No. 451-03-68/2020-14/200026).

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ЗАГАЂЕЊЕ СЕДИМЕНАТА ЖИВОМ У РЕЦИ ТИСИ (СРБИЈА)

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РЕЗИМЕ: Велики број међународних организација хемијски елемент живу (Hg) означио је као глобално загађујућу супстанцу високог приоритета због своје мобилности и перзистентности у животној средини, као и високе токсичности за живе организме. Истраживање је спроведено с циљем да се утврди: (i) укупни садржај Hg (THg) и њена просторна дистрибуција у седиментима дуж тока реке Тисе, (ii) могући извори THg и (iii) степен загађења седимената реке Тисе примењујући различите критеријуме. Укупан садржај Hg у седиментима реке Тисе кретао се од 0,07 до 0,49 mg kg^{-1}, са средњом вредношћу ± S.D. од 0,26 ± 0,10 mg kg^{-1}. Највећа средња вредност THg (0,30 mg kg^{-1}) утврђена је у доњем току реке, док је најнижа (0,13 mg kg^{-1}) забележена у притокама. На основу анализе главних компонената (PCA) може се закључити да оптерећење седимената металима дуж целог тока реке Тисе има заједничко порекло. Оно се доводи у везу са антропогеним активностима на основу израчунатих вредности фактора обогаћивања (EF). Укупни садржаји Hg у седиментима реке Тисе виши су од природне вредности за тај део слива, али су у границама максимално дозвољених концентрација према званичном стандарду Републике Србије. У поређењу са националним смерницама за океанску и атмосферску управу (NOAA) у 80,49% узорака седимената THg представља минималан и могући ризик за организме. Интегришући резултате процене загађења, може се закључити да THg у седиментима реке Тисе у Србији показује знатну контаминацију на основу геоакумулационог индекса (Igeo) и фактора загађења (CF), као и висок ризик загађења на основу индекса потенцијалног ризика на животну средину (PERI).

КЉУЧНЕ РЕЧИ: жива (Hg), процена ризика, седименти, река Тиса