



Potentially toxic elements in the riparian soils of the Sava River

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Abstract

Purpose Riparian zone contamination is a growing problem for several European catchments due to high anthropogenic pressures. This study investigates As, Cd, Cr, Cu, Ni, Pb, and Zn concentrations in the Sava River riparian zone, characterized by wide agricultural areas, various geological substrates, and different types of industrial pollution. The accumulation and mobility of these elements were studied because they are listed as priority substances in the Water Framework Directive and environmental objectives for surface waters.

Materials and methods Sampling was performed during the sampling campaign of the EU 7th FW-funded GLOBAQUA project in September 2015 during a low-water event. Soil samples were collected along the Sava River at 12 selected sampling sites, from a depth of 0–30 cm, at a distance of 10–15 m from the river bank. The extent of pollution was estimated by determining total and readily soluble element concentrations in the soils. Potential ecological risk and the source of the selected elements in the soils was determined using the enrichment factor (EF), potential ecological risk index (RI), and statistical methods such as the principal component analysis (PCA) and multiple linear regression analysis (MLRA).

Results and discussion This study showed that concentrations of the selected elements increase along the Sava. In terms of origin, PCA and MLRA indicated that Cr and Ni in soils are predominantly lithogenic, while As, Cd, Pb, and Zn are both lithogenic and anthropogenic (ore deposits, industry, and agriculture). PCA singled out Cu since its origin in soil is most probably from specific point-source pollution. EF was generally minor to moderate for most of the examined elements, apart from Cu, for which the EF was significant at one sampling site. Overall ecological risk (RI) fell within the low-risk category for most sites, apart from Belgrade sampling site (BEO), where high total Cd content affected individual and overall ecological risk indicators, indicating Cd could represent a considerable ecological risk for the downstream riparian zone.

Conclusions At downstream sites, there was a noticeable increase in PTE content, with Cd, Cr, Ni, and Zn exceeding the proposed threshold values for European soils, indicating rising contamination in riparian soils. In terms of the ecological risk, only Cd could pose a potential ecological threat for the downstream riparian zone.

Keywords Ecological risk index · Enrichment factor · Potentially toxic elements · Riparian soil · The Sava River

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1 Introduction

Riparian zones are unique, dynamic systems whose size is dependent on the topography. Often characterized by high biodiversity thanks to the heterogeneous environment created by frequent flooding, fluctuations in the groundwater system, and deposition of sediments (Naiman and Décamps 1997), their boundaries can vary greatly. Generally, riparian zones extend outwards from the river channel to the limits of flooding and travel into the canopy of riverside vegetation. They play a key role in the functioning of aquatic ecosystems, affecting chemical, physical, and biological processes (Sedell et al. 1991; Naiman and Décamps 1997; Pusey and Arthington 2003). Riverine riparian areas influence chemical

loads from diffuse industrial and agricultural sources (runoff) and reduce in-stream pollution during flooding (Karr and Gorman 1975), while riparian soil acts as an important sink for pollutants, especially heavy metals from river water or upland, through adsorption and sedimentation (Zhang et al. 2010; Izquierdo et al. 2013). These soils are under intense pressure from various anthropogenic sources of pollutants, which could contribute significantly to multiple stress conditions (Navarro-Ortega et al. 2015; Wang et al. 2015). In fact, as a complex and dynamic component of aquatic ecosystems, riparian soils are a suitable medium for monitoring heavy metal pollution.

Past research has paid relatively little attention to wetland soils, especially floodplain soils, despite them frequently having high levels of pollutants (Rinklebe et al. 2007), and with the progressive development of riverbanks in urban areas, on farmland, and in industrial zones, contamination problems will only increase markedly in the years to come (Saint-Laurent et al. 2013). Among various pollutants, chemical elements may contribute significantly to the overall pollution of riverine environments. Most often, their origin is lithogenic, although anthropogenic activities may increase concentrations and lead to contamination (Bain et al. 2012; Jiang et al. 2017). Unlike the former, elements of anthropogenic origin are highly mobile and bioavailable, and thus more likely to impact negatively on aquatic ecosystems (Tessier and Campbell 1987). To assess the origin of heavy metals in soils, multivariate statistical analyses, like principal component analysis (PCA), are often performed (Borůvka et al. 2005; Cakmak et al. 2015; Jiang et al. 2017), while the enrichment factor (EF) and ecological risk index (RI) are frequently used to assess the ecological risk potentially toxic elements (PTE) can pose to riverine environments (Hakanson 1980; Hu et al. 2013; Sakan et al. 2015; Čakmak et al. 2018).

Flowing through four countries (Slovenia, Croatia, Bosnia and Herzegovina, and Serbia), the Sava River is the major drainage basin (97,713.20 km²) of Southeastern Europe and the largest tributary to the River Danube (ISRBC 2013). The Sava River Basin (SRB) is characterized by high biological diversity and industrial and economic potential. The Sava is also one of the main sources of drinking water for its riparian population. Sources of PTE in the Sava's riparian zones are diverse, relating mostly to chemical weathering of parent material and various anthropogenic factors (Vertačnik et al. 1995; Orescanin et al. 2004; Crnković et al. 2008; Markovics et al. 2010; Vuković et al. 2011; Šajn and Gosar 2014; Ščančar et al. 2015). These factors include industrial and intensive agricultural activities, urbanization, and, in the Sava's lower reaches, non-treated municipal sewage (Komatina and Grošelj 2015). Contaminants from diffuse and point sources have a major impact on water quality and sediments (Ščančar et al. 2015), while hydrological conditions significantly impact pollution levels. Namely, during flooding, water covers the riparian soil

and if this is contaminated with PTE, these may be released into the overlying water and accumulate in sediments (Milačić et al. 2017). Due to the persistence of many pollutants and their bioaccumulation potential, sediments represent a serious threat to riverine ecosystems (Vuković et al. 2011). Studies on heavy metals and other pollutants in river water and sediments have multiplied in recent years, especially for large rivers such as the Danube (Woitke et al. 2003; Crnković et al. 2008; Pavlović et al. 2016), the Sava and their tributaries the Drava and the Kolubara (Orescanin et al. 2004; Crnković et al. 2008; Vuković et al. 2011; Vidmar et al. 2017; Milačić et al. 2017; Čakmak et al. 2018), the Tisza (Sakan and Đorđević 2010), and the Po (Vignati et al. 2003). Other researchers have studied contamination of riparian soil along the Danube (Pavlović et al. 2016), the Sava and their tributaries the Drava and the Kolubara (Vertačnik et al. 1995; Halamić et al. 2003; Šajn et al. 2011; Šajn and Gosar 2014; Čakmak et al. 2018), and the Odra (Ciesielczuk et al. 2014), but such studies are documented much scarcely.

The aim of the present study was to investigate the extent of soil contamination by PTE (As, Cd, Cr, Cu, Ni, Pb, and Zn) in the Sava's riparian zone, to identify sources of these elements in riparian soil, and to assess the ecological risk PTE may pose to such soil. Trace elements were selected in line with the list of priority substances in the field of water policy within the Water Framework Directive (WFD) and its daughter directives: Directive 2000/60/EC (EC 2000) and Directive 2013/39/EU (EU 2013).

2 Material and methods

2.1 Study area and sampling

The study area covered the Sava's entire 945 km, from its source (Sava Dolinka) to its confluence with the Danube, with sampling sites selected in Slovenia, Croatia, and Serbia. Characterized by a diverse landscape, the SRB can be roughly divided into three parts—upper, middle, and lowland. The 265-km upper reach, including Sava Dolinka, is dominated by mountainous terrain, the shortest middle part (129 km) flows through lowland, and the 597-km lower section flows through plains and is characterized by a very low declination and a wide water channel (ISRBC 2013). Specific hydrological and soil characteristics have resulted in vegetation diversity, including 167 protected areas, and while riparian soils are rich in organic residues, the humus content is normally low because these residues are covered by river sediment during high-water events, preventing decomposition (Karadžić et al. 2015).

Sampling was conducted along the river during a low-water event from 1 to 9 September 2015 as part of the GLOBAQUA project (Navarro-Ortega et al. 2015). Twelve sampling

localities were selected based on their representativeness in terms of different pollution sources/anthropogenic pressures (Fig. 1). The sampling sites' main characteristics are presented in Table S1 (Electronic Supplementary Material). Soil samples were taken at each site from a depth of 0–30 cm so as to cover both lithogenic and anthropogenic element sources and at a distance of 10–15 m from the river bank because this area usually floods during high-water events and is thus prone to the effects of pollutants from water and sediments. Five sub-samples per sampling site were combined into one composite sample, which was transferred to PVC buckets and thoroughly homogenized before further treatment. Samples were kept in the dark at 4 °C before being analyzed for trace element content. All analyses were performed in five replicates and the results expressed on a dry mass basis.

2.2 Instrumentation and reagents

Microwave digestion of soil samples was performed on a CEM Corporation (Matthews, NC, USA) MARS 5 Microwave System. Element concentrations in digested samples and soil fractions extracted in 0.11 mol L⁻¹ acetic acid were determined using inductively coupled plasma mass spectrometry (ICP-MS) (7700×, Agilent Technologies, Tokyo, Japan). For sample preparation, suprapure acids (Merck, Darmstadt, Germany) and Milli-Q water (Direct-Q 5 Ultrapure water system, 18 MΩ cm, Millipore Watertown, MA, USA) were used, while other chemicals used were of analytical reagent grade or higher purity. ICP multi-element standard solution IV CertiPUR containing 1000 mg L⁻¹ ± 10 mg L⁻¹ in 1 mol L⁻¹ HNO₃ of different elements was obtained from Merck. Samples were filtered using 0.45-μm Minisart cellulose nitrate membrane filters (Sartorius, Goettingen, Germany).

2.3 Quality control

Certified reference materials SPS-SW1 Quality Control Material for Surface Water Analysis from SPS Spectrapure Standards AS (Oslo, Norway) and CRM 320R Trace Elements in River Sediment from the Community Bureau of Reference (Geel, Belgium) were used to check accuracy. The data presented in Table S2 (Electronic Supplementary Material) correlated closely with the reported certified values (better than ± 5%), thus confirming the accuracy of the analytical procedures applied.

2.4 Determination of total element concentrations in soil

To determine total element concentrations, approximately 0.25 g of soil was transferred to Teflon vessels and 4 mL HNO₃ (s.p.), 2 mL HF (s.p.), and 1 mL HCl (s.p.) were added.

The vessels were covered and subjected to microwave-assisted digestion by ramping the temperature to 200 °C in 30 min, maintaining the temperature for 60 min and then cooling the contents over the next 30 min. 12.5 mL H₃BO₃ (s.p.) (4% aqueous solution) was then added for complexation of fluorides and the contents were digested again by ramping the temperature to 200 °C in 15 min, maintaining the temperature for 30 min and then cooling the contents over the next 30 min (Ščančar et al. 2007). After digestion, clear solutions were obtained. The contents were transferred quantitatively to 30-mL graduated polypropylene tubes and filled to the mark with water. The same procedure, but minus the soil samples, was applied to determine blanks. Element concentrations in digested soil samples were determined by ICP-MS.

2.5 Determination of readily soluble element concentrations in 0.11 mol L⁻¹ acetic acid

To determine readily soluble, potentially bioavailable element concentrations, acetic acid extraction was applied (Quevauviller et al. 1997; Milačič et al. 2010). Approximately 2 g of moist soil sample was weighed into a 40-mL centrifuge tube with 20 mL 0.11 mol L⁻¹ acetic acid added. Samples were shaken on a mechanical shaker (300 rpm) for 16 h, centrifuged (9000 rpm, 20 min), and filtered through a 0.45-μm membrane filter. Element concentrations were determined by ICP-MS.

2.6 Calculation of the enrichment factor and ecological risk index

A geochemical approach, such as the enrichment factor (EF) and ecological risk index (RI), has been widely used to assess the environmental burden of elements accumulated in soils and sediments (Hu et al. 2013; Sakan et al. 2015; Čakmak et al. 2018). The EF was calculated according to the following equation:

$$EF = \frac{(C_x/C_{Mn})_{\text{soil}}}{(C_x/C_{Mn})_{\text{background}}}$$

where $(C_x/C_{Mn})_{\text{soil}}$ is the ratio between concentrations of the potentially enriched element C_x and Mn concentrations (C_{Mn}) in soil samples, and $(C_x/C_{Mn})_{\text{background}}$ is the ratio of the referenced background values. Manganese was used as the reference background element due to a lack of Al data, with its feasibility confirmed by Loska et al. (1997), as Mn is mainly of lithogenic origin and its anthropogenic sources are minimal. Principal component analysis (PCA) can assist when selecting the reference background element (Birch 2017), as can a high correlation with the elements studied (N'guessan et al. 2009). In our study, using Mn as the reference metal was justified through PCA, which identified it as being of lithogenic origin and in significant correlation with most of

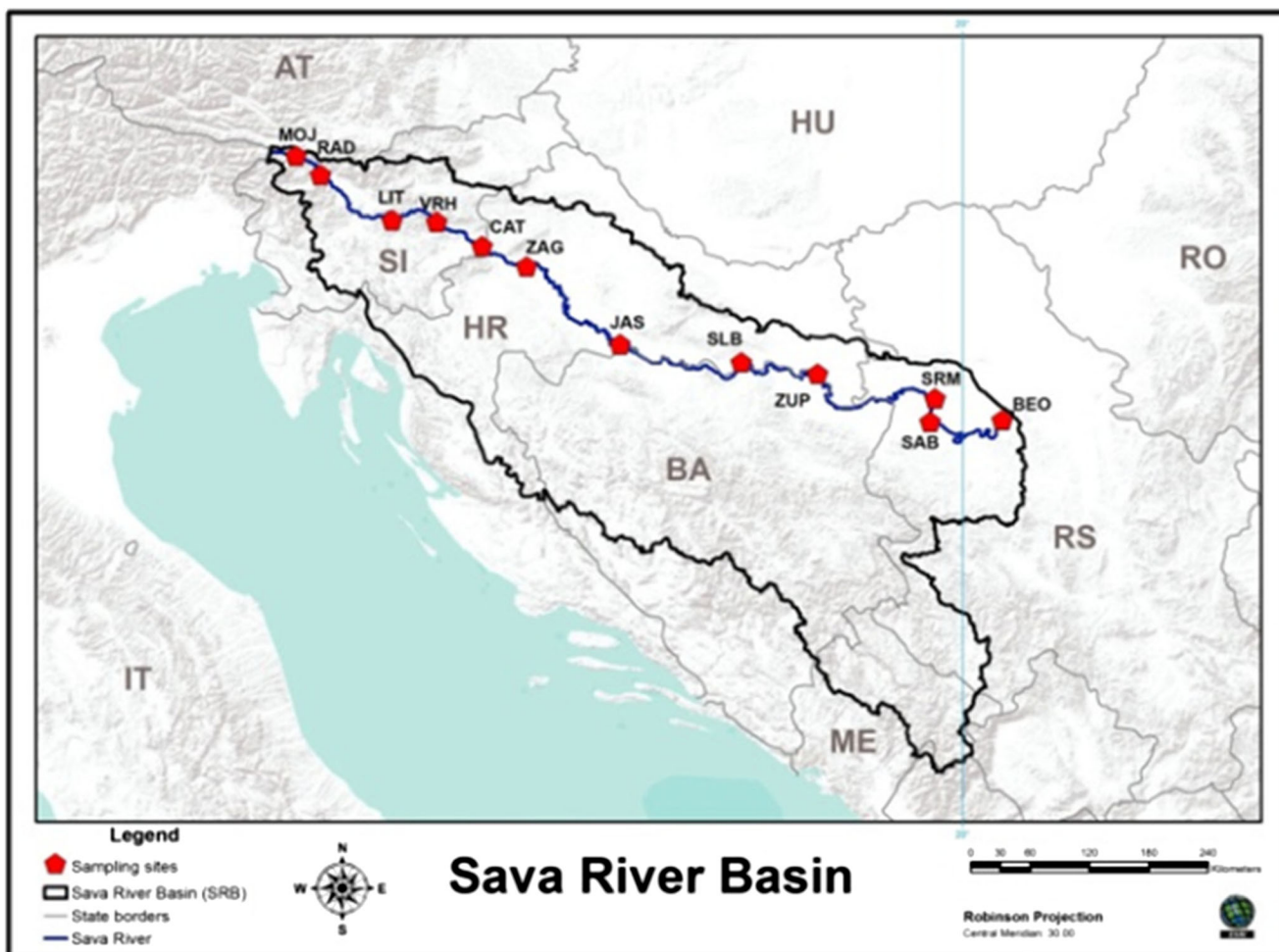


Fig. 1 Sava River sampling sites (sampling in September 2015): 1, Mojstrana (SI) MOJ; 2, Radovljica (SI) RAD; 3, Litija (SI) LIT; 4, Vrhovo (SI) VRH; 5, Čatež (SI) CAT; 6, Zagreb (HR) ZAG; 7,

Jasenovac (HR) JAS; 8, Slavonski Brod (HR) SLB; 9, Županja (HR) ZUP; 10, Sremska Mitrovica (RS) SRM; 11, Šabac (RS) SAB; 12, Belgrade (RS) BEO

the studied elements, in particular with Cr and Ni. Five contamination categories are recognized on the basis of the enrichment factor (Sutherland 2000): $EF < 2$ represents deficiency to minimal enrichment; $EF 2-5$, moderate enrichment; $EF 5-20$, significant enrichment; $EF 20-40$, very high enrichment; and $EF > 40$, extremely high enrichment.

The potential ecological risk index (RI) takes into account the concentration effects of various trace metals in soils, the toxic effects of different metals, and the synergistic effects of multiple element complex pollution as defined by Hakanson (1980). The RI was calculated as the sum of pollutant risk factors, using the equation:

$$RI = \sum E_i$$

where E_i represents a single risk factor for trace element i and is defined as:

$$E_i = T_i f_i = T_i \frac{C_i}{B_i}$$

where T_i is the toxic response for trace element i . The T_i values for Cd, As, Ni, Cu, Pb, Cr, and Zn are 30, 10, 5, 5, 5, 2, and 1 respectively. f_i is the ratio of the measured concentration C_i to the calculated background concentration B_i in soils. The potential ecological risk of trace elements in soils was classified into five categories based on E_i and four categories based on RI (Hakanson 1980): $E_i < 40$ represents low potential ecological risk; $40 \leq E_i < 80$, moderate potential ecological risk; $80 \leq E_i < 160$, considerable potential ecological risk; $160 \leq E_i < 320$, high potential ecological risk; and $E_i \geq 320$, very high potential ecological risk; while $RI < 150$ represents low risk; $150 \leq RI < 300$, moderate risk; $300 \leq RI < 600$, considerable risk; and $RI \geq 600$, very high risk.

2.7 Statistical analyses

To assess the origin (natural vs. anthropogenic) of the studied elements, multivariate analyses, such as the Pearson correlation, PCA, and multiple linear regression analysis (MLRA), were used. Descriptive and multivariate statistical analyses

were performed using MS Excel 2010 and SPSS version 20 software (SPSS, v21 2009). The arithmetical median method was used to calculate toxic element background values for soils in Serbia (Mrvić et al. 2011), Slovenia (Slovenian Environment Agency 2017), and Croatia (Halamić et al. 2012).

3 Results

3.1 Analysis of trace element concentrations in riparian soils

Total and readily soluble As, Cd, Cr, Cu, Ni, Pb, and Zn concentrations in soils were determined to evaluate the extent of pollution in the Sava River riparian zone. Data on total element concentrations and calculated background values for the SRB are listed in Table 1 with results representing the average of 5 independent analyses. Deviation from the average value for all elements did not exceed $\pm 5\%$.

PTE concentrations varied among the sampling localities. In the upper and middle reaches, total concentrations were within the average values for European soils (Gawlik and Bidoglio 2006), while an increase in element concentrations was observed in the lower reaches. As, Cu, and Zn concentrations exceeded calculated background values at almost all the sampling sites, Cr and Ni in the lower stretch, while Cd and Pb concentrations fell mainly within the calculated background values (Table 1).

The level of potentially readily soluble element concentrations versus their total concentrations in soils is shown in Table 2. The levels of the readily soluble fractions are generally low for As, Cr, Cu, and Pb (below 1%) and for Ni and Zn (below 4%), while they are higher for Cd (3–40%).

3.2 Source identification and ecological risk assessment of trace elements

In our study, the Pearson correlation was applied to find the relationship between the studied elements. As Table S3 (Electronic Supplementary Material) shows, there was a significant positive correlation between the elements, apart from Cu. PCA was applied to identify trace element sources in the riparian soil and showed three principal components (PC1, PC2, and PC3), which explained 95.03% of the total variability. PC1 included 67.75% of total variability; PC2, 14.22%; and PC3, 13.06%. PC1 corresponds to natural (lithogenic) factors, PC2 is based on the variability of elements controlled by natural factors such as the geological substrate and anthropogenic inputs, while PC3 relates to anthropogenic factors (Fig. 2 and Table S4, Electronic Supplementary Material). PC1 indicated the common, predominantly lithogenic origin of Cr, Mn, and Ni, with their influence seen largely downstream (at SLB, SAB, ZUP, and SRM) (Fig. 2a, b). PC2 is

mainly impacted by Cd, Pb, and Zn, indicating both their geogenic and anthropogenic origin (Fig. 2a). Point-source pollution with the highest value in PC2 was observed for Pb, which could be explained by anthropogenic factors at BEO, but also by the impact of chemical weathering and ore deposits, which is shown at MOJ (Fig. 2b). Arsenic was also shown to occur in both PC1 and PC2 with equally strong bonds, indicating both its anthropogenic and lithogenic origins, depending on the sampling site. The differentiation of Cu from the other components indicated an anthropogenic pollution source, which is highly marked at CAT, with the impact transferred downstream to ZAG and JAS (Fig. 2b). MLRA indicated the dominant influence of a factor that does not belong to the main components for all elements except Cu, Cr, and Ni. In the Sava's case, this could refer to frequent water level fluctuations and sediment deposition on the soil's surface layers (Fig. 3).

The EF was calculated for all sites and generally, the order of average values was $Cu > Zn > Cr > Ni > As > Pb > Cd$ (Table 3). Values ranged from minor to moderate enrichment, depending on the site, and were only in the significant range for Cu at CAT. The mean E_i calculated for all sites can be ranked in the following order: $Cd > As > Cu > Ni > Pb > Cr > Zn$, with Cd having the highest value (32.27) and Zn the lowest (1.80). Individual E_i values were mostly in the low-risk category, apart from Cd in the river's lower reaches (at SRM, SAB, and BEO), where it ranged from moderate to considerable. The RI was calculated for all sites, with the highest value at BEO (215.2) and the lowest at LIT (28.17). Moderate RI was observed only at BEO (Table 4).

4 Discussion

Due to their unique semi-terrestrial position, riparian zones are remarkably sensitive to alterations in flow, temperature, landscape composition, and anthropogenic pressures. PTE enter these zones as a result of the weathering of parent rocks and anthropogenic activities, as well as floods, and are incorporated into sediments and soils as pollutants (Xie et al. 2014; Milačić et al. 2017). With PTE concentrations in rivers increasing markedly in Europe during the nineteenth and twentieth centuries (Schulz-Zunkel et al. 2013), determining their levels in natural waters and sediments is of great importance, so as to investigate the geochemical migration of chemical species and also substantiate environmental pollution in aquatic systems due to human activities (Vertačnik and Bišćan 1993). Multivariate statistics have proven to be a powerful tool for identifying geogenic and anthropogenic groups of chemical elements, which is vital as the latter are more mobile and bioavailable (Šajn and Gosar 2014).

According to Alloway (2013), average As concentrations in soils range from 5 to 7.5 $mg\ kg^{-1}$, while there is no data on

Table 1 Total content of potentially toxic elements in soils from the Sava River riparian zone. The values are mean (SD)

Sampling sites	Elements (mg kg ⁻¹ dry weight)						
	As	Cd	Cu	Cr	Ni	Pb	Zn
MOJ	10.57 (0.96)	0.18 (0.02)	17.91 (0.85)	36.28 (0.98)	15.60 (0.59)	78.14 (1.46)	180.13 (5.13)
RAD	7.59 (0.64)	0.23 (0.02)	32.73 (1.23)	60.65 (1.96)	27.36 (1.61)	45.74 (1.37)	182.01 (3.91)
LIT	9.32 (0.74)	0.13 (0.01)	31.17 (0.78)	42.53 (1.25)	22.88 (1.21)	27.61 (0.76)	91.67 (4.09)
VRH	11.25 (0.55)	0.27 (0.03)	26.69 (0.76)	62.14 (1.81)	28.93 (1.59)	31.64 (0.83)	109.26 (2.55)
CAT	12.70 (0.94)	0.62 (0.06)	148.62 (1.79)	77.96 (2.01)	36.81 (1.71)	43.94 (0.64)	170.38 (3.53)
ZAG	8.68 (0.57)	0.31 (0.03)	65.02 (1.53)	52.16 (1.83)	24.47 (1.37)	21.07 (0.44)	114.48 (2.15)
JAS	8.26 (0.66)	0.18 (0.02)	67.71 (1.84)	38.07 (1.25)	22.40 (1.14)	22.50 (0.45)	104.44 (3.00)
SLB	13.89 (0.90)	0.31 (0.03)	39.28 (1.12)	161.55 (3.57)	85.83 (4.12)	25.77 (0.70)	109.92 (4.72)
ZUP	16.18 (0.89)	0.38 (0.04)	51.31 (1.97)	282.79 (5.83)	165.73 (12.16)	42.99 (1.36)	170.99 (5.28)
SRM	16.30 (0.63)	1.44 (0.14)	42.7 (0.87)	265.11 (12.36)	122.68 (5.93)	50.91 (0.95)	192.86(6.06)
SAB	21.29 (1.36)	1.61 (0.16)	41.33 (1.49)	235.85 (7.01)	152.60 (7.89)	58.78 (1.77)	224.61 (8.70)
BEO	29.33 (1.87)	3.11 (0.31)	63.74 (1.28)	221.67 (5.16)	168.04 (6.89)	83.60 (2.12)	328.29 (6.86)
Calculated background values	11.53	0.68	24.12	72.52	41.33	44.03	91.64
Background values for European soils ^a	–	1–3	50–140	50–100	30–75	50–300	150–300

^a Proposed threshold values for European soils according to Directive 86/278/EEC (Gawlik and Bidoglio 2006)

background As values for European soils. In this study, As concentrations ranged from 7.6 to 29.3 mg kg⁻¹ (Table 1) and were generally lower than those found in the alluvial soils of the Drava (7–54 mg kg⁻¹, Šajn et al. 2011), but higher than those reported for the Sava in earlier studies (5–18 mg kg⁻¹, Halamić et al. 2003). With As affecting both PC1 and PC2, its origin in the Sava’s riparian soil could be both lithogenic and anthropogenic, which is supported by As correlating closely with Mn, Cr, and Ni and with Pb, Zn, and Cd (Table S3, Electronic Supplementary Material). In the river’s upper reaches, EF values showed moderate enrichment, indicating mixed geogenic and anthropogenic influences, which most likely originate from ore deposits and the steel industry at Jesenice (Vidmar et al. 2017) (Fig. 2a). In the lower reaches, total As concentrations increase, while the potentially available portion of As decreases. Here, EF values pointed to the mainly lithogenic origin of As at SLB, ZUP, and SAB, but its anthropogenic origin at BEO (Fig. 2b).

In the present study, total Cd concentrations increased along the Sava, varying from 0.13 mg kg⁻¹ at LIT to 3.11 mg kg⁻¹ at BEO. Levels were mostly below background values for European soils (1–3 mg kg⁻¹) (Gawlik and Bidoglio 2006), except at BEO (Table 1), and were similar to those reported in a previous study on the Sava (Vertačnik et al. 1995), but lower than those at the Drava (0.10–17 mg kg⁻¹, Šajn et al. 2011). The low total concentrations indicate that Cd does not pose an ecological risk, a fact supported by the minimal enrichment EF values (Tables 3 and 4). However, downstream, at SAB and BEO, total concentrations were higher (> 1 mg kg⁻¹, Table 1), most probably due to anthropogenic

sources (Fig. 2a, b), such as untreated sewage water, and the chemical and metal industries (Milačić et al. 2017). Its mixed origin in soil was highlighted by PCA (Cd extracted in PC2) and its close correlation with As, Cr, Ni, Pb, and Zn (Table S3, Electronic Supplementary Material; Figs. 2 and 3). These high Cd concentrations are also a result of the reduction of river flow, leading to Cd accumulation. Levels of bioavailable Cd decreased with distance downstream with > 20% in the upper reaches (Table 2) due to its high solubility (Mitchell et al. 2016) and < 10% in the lower stretch as it probably becomes

Table 2 Readily soluble concentrations of the investigated elements in soils in the Sava River riparian zone

Sampling sites	Readily soluble concentrations (%)						
	As	Cd	Cu	Cr	Ni	Pb	Zn
MOJ	1.05	38.19	0.21	0.04	0.09	0.05	3.34
RAD	0.13	25.36	0.21	0.06	2.75	0.09	3.40
LIT	0.12	19.49	0.23	0.01	1.47	0.02	1.88
VRH	0.09	17.13	0.23	<LoQ	1.11	0.01	1.98
CAT	0.12	10.06	0.31	0.02	0.90	<LoQ	2.14
ZAG	0.21	6.96	0.05	0.01	0.58	<LoQ	0.62
JAS	0.14	24.96	0.05	0.01	1.04	<LoQ	1.99
SLB	0.11	7.09	0.07	0.00	0.33	<LoQ	0.25
ZUP	0.18	9.21	0.06	0.00	0.44	<LoQ	0.46
SRM	0.18	4.23	0.09	0.00	0.56	<LoQ	0.57
SAB	0.17	3.09	0.07	0.01	0.37	<LoQ	0.55
BEO	0.15	5.76	0.10	0.00	0.45	<LoQ	0.73

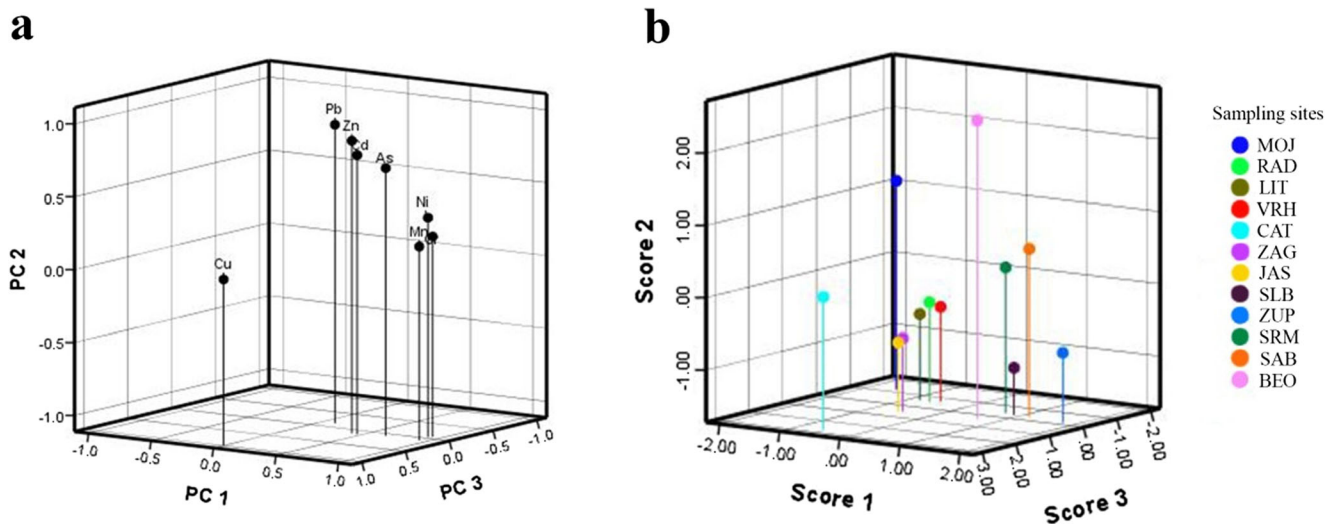


Fig. 2 **a** PCA loading plot for soil samples in the riparian zone of the Sava River. **b** Graphic score for soil samples in the riparian zone of the Sava River for 12 sampling sites (MOJ, RAD, LIT, VRH, CAT, ZAG, JAS, SLB, ZUP, SRM, SAB, and BEO)

bound due to various more sparingly soluble chemical phases (Ashraf et al. 2012; Mitchell et al. 2016). Higher than the proposed threshold values, the total Cd content impacted on a particularly high E_i (considerable potential ecological risk) and, as such, on the overall RI (Table 4). Hence, due to its pronounced toxicity and since Cd comprises at least 50% of the overall RI, it can potentially represent an ecological risk in the Sava's lower reaches (Table S5, Electronic Supplementary Material).

Copper showed considerable concentration variations, from 18 to 149 mg kg⁻¹ (Table 1). At all sampling sites, concentrations are lower in relation to background values

for European soils (50–140 mg kg⁻¹, Gawlik and Bidoglio 2006), except at CAT. Generally, our study revealed higher concentrations than those from earlier studies on the riparian soils of the Sava (12–35 mg kg⁻¹, Halamić et al. 2003) and the Odra (0.44–75.29 mg kg⁻¹, Ciesielczuk et al. 2014). Individual and localized pollution resulted in the differentiation of Cu from other components in the PCA, indicating a specific anthropogenic source of pollution (Fig. 2a, b), a fact supported by MLRA (Fig. 3). EF levels revealed minor enrichment, apart from at RAD, LIT, ZAG, and JAS (moderate enrichment), (Table 3). At CAT, significant enrichment was found, probably due to industrial activities (the paper and

Fig. 3 Apportionment percentage of possible source types (lithogenic, mixed lithogenic and anthropogenic, anthropogenic, other) based on observed PTE concentrations (MLR analysis)

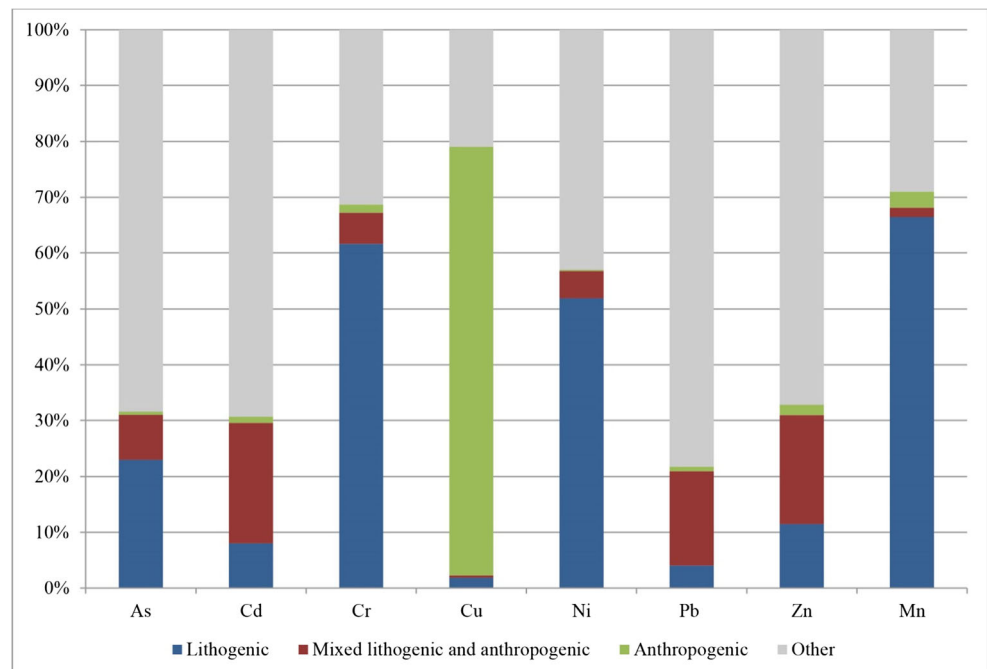


Table 3 Enrichment factors (EF) for soils in the Sava River riparian zone

Sampling sites	Enrichment factor (EF)						
	As	Cd	Cu	Cr	Ni	Pb	Zn
MOJ	2.31	0.67	1.87	1.26	0.95	4.48	4.96
RAD	1.10	0.55	2.26	1.39	1.10	1.73	3.31
LIT	2.69	0.62	4.30	1.95	1.84	2.09	3.33
VRH	1.70	0.70	1.93	1.50	1.22	1.26	2.08
CAT	1.40	1.17	7.86	1.37	1.14	1.27	2.37
ZAG	1.27	0.78	4.56	1.22	1.00	0.81	2.11
JAS	1.15	0.43	4.51	0.84	0.87	0.82	1.83
SLB	0.93	0.35	1.26	1.72	1.61	0.45	0.93
ZUP	0.87	0.35	1.32	2.42	2.49	0.61	1.16
SRM	1.50	2.26	1.89	3.89	3.16	1.23	2.24
SAB	1.25	1.61	1.16	2.21	2.51	0.91	1.66
BEO	1.76	3.17	1.83	2.12	2.82	1.32	2.49
Min	0.87	0.35	1.16	0.84	0.87	0.45	0.93
Max	2.69	3.17	7.86	3.89	3.16	4.48	4.96
Mean	1.50	1.05	2.90	1.83	1.73	1.41	2.37

wood processing industry, ISRBC 2016). In addition, frequent Cu use in agriculture and different metallurgical processes, such as the production of steel and steel products, can also be a source of soil contamination (Alloway 2013). Although EF indicated significant enrichment, potential ecological risk (RI) was low, indicating that Cu does not pose an ecological risk in that area (Table 4 and Table S5, Electronic Supplementary Material).

Total Cr soil concentrations, ranging from 36.3 mg kg⁻¹ (at MOJ) to 282.8 mg kg⁻¹ (at ZUP), were similar to those determined in alluvial soil at the Drava (40–250 mg kg⁻¹, Šajin et al.

2011), but higher than those found in earlier studies on the Sava (Vertačnik et al. 1995). Ni soil concentrations, ranging from 15.6 to 168 mg kg⁻¹, were similar to those reported for the Kolubara (17.9–178.4 mg kg⁻¹, Čakmak et al. 2018), but were higher than those at the Drava (18–61 mg kg⁻¹, Šajin et al. 2011) and the Odra River (0.90–16.13 mg kg⁻¹; Ciesielczuk et al. 2014). From MOJ down to JAS, total Cr and Ni content was in line with average concentrations for European soils (50–100 mg kg⁻¹ for Cr; 30–75 mg kg⁻¹ for Ni, Gawlik and Bidoglio 2006). However, from JAS to BEO, levels increased and exceeded threshold values (Table 1). PCA and MLRA showed that Cr and Ni are both lithogenic in origin (Figs. 2 and 3), mainly from the weathering of parent materials, particularly mafic and ultramafic rocks (Alloway 2013), with the Dinaric Ophiolite Belt contributing to elevated Cr and Ni concentrations in the SRB (Mrvić et al. 2011; Grba et al. 2015). In the lower reaches, from ZUP to BEO, the geological substrate has the greatest impact on Cr and Ni soil content, which is also affected by erosion and fluvial processes from the Drina and Kolubara (Belanović-Simić et al. 2013; Čakmak et al. 2018) (Fig. 2a, b). However, elevated concentrations also arise from local anthropogenic pollution sources (Milačić et al. 2017), as shown by the slightly elevated, moderate enrichment levels in all but the upper reaches (minor enrichment) (Table 3). However, despite the high Cr and Ni concentrations in the Sava’s lower stretch, their impact on RI is insignificant (Table 4).

Total Pb soil concentrations, ranging from 21 to 83.6 mg kg⁻¹, were similar to those reported in a previous Sava study (18–58 mg kg⁻¹, Halamić et al. 2003), but higher than at the Kolubara (8.8–24.5 mg kg⁻¹, Čakmak et al. 2018) and Odra (0.96–22.01 mg kg⁻¹, Ciesielczuk et al. 2014), yet still within the proposed background values for European soils (50–300 mg kg⁻¹) at all localities (Table 1). In general, Pb

Table 4 Ecological risk index for individual elements (*E_i*) and the potential ecological risk index (RI) for the environment for soils in the Sava River riparian zone

Sampling sites	Individual ecological risk index (<i>E_i</i>)							RI
	As	Cd	Cu	Cr	Ni	Pb	Zn	
MOJ	9.17	7.94	3.71	1.00	1.89	8.87	1.97	34.55
RAD	6.58	9.93	6.79	1.67	3.31	5.19	1.99	35.46
LIT	8.09	5.55	6.46	1.17	2.77	3.14	1.00	28.17
VRH	9.75	12.08	5.53	1.71	3.50	3.59	1.19	37.37
CAT	11.01	27.45	30.81	2.15	4.45	4.99	1.86	82.72
ZAG	7.53	13.90	13.48	1.44	2.96	2.39	1.25	42.94
JAS	7.16	8.07	14.04	1.05	2.71	2.56	1.14	36.72
SLB	12.05	13.60	8.14	4.46	10.38	2.93	1.20	52.76
ZUP	14.03	16.85	10.64	7.80	20.05	4.88	1.87	76.11
SRM	14.14	63.66	8.86	7.31	14.84	5.78	2.10	116.69
SAB	18.46	70.99	8.57	6.50	18.46	6.68	2.45	132.11
BEO	25.44	137.25	13.21	6.11	20.33	9.49	3.58	215.42
Mean	11.95	32.27	10.85	3.53	8.80	5.04	1.80	

concentrations were consistent along the Sava, apart from at MOJ, where higher concentrations could be due to the chemical weathering of parent material and ore deposits, and at BEO, due to upstream industrial activities. Lead correlates closely with As, Cd, and Zn, which are extracted in a separate group in PC2 (Fig. 2a, b). Indeed, Pb accumulates in soil in the surface horizons, mostly from anthropogenic sources (Alloway 2013). However, the EF for Pb was generally low, again with the exception of MOJ (moderate EF), and while point and diffuse sources of Pb pollution, such as industrial activities, mining and smelting, and leaded gasoline emissions, pose a high environmental risk, in this study, potentially bioavailable concentrations were mostly below the limit of quantification ($< \text{LoQ}$) and E_i was low, indicating that Pb does not present an environmental risk (Tables 2 and 4).

Total Zn concentrations, ranging from 91.7 mg kg^{-1} (LIT) to 328.3 mg kg^{-1} (BEO), were within the values for European soils ($150\text{--}300 \text{ mg kg}^{-1}$; Gawlik and Bidoglio 2006) at all localities except BEO (Table 1). While levels were similar to an earlier Sava study by Vertačnik et al. (1995), they were higher than those in a second study ($53\text{--}135 \text{ mg kg}^{-1}$, Halamić et al. 2003) and another on the Kolubara ($36\text{--}126 \text{ mg kg}^{-1}$, Čakmak et al. 2018). Zinc in soils mainly depends on the type of parent material; however, apart from geological processes, Zn sources are generally related to anthropogenic activities, such as mining, smelting, and the application of different fertilizers (Adriano 2001; Alloway 2013). In this study, PCA showed that Zn concentrations are mostly associated with PC2 (Fig. 2a, b), with it correlating closely with As, Cd, and Pb (Table S3, Electronic Supplementary Material). Downstream, from ZUP to BEO, Zn levels increased noticeably, particularly at BEO, with the source potentially being untreated sewage waters (Milačić et al. 2017; Adamiec et al. 2016). The EF for Zn was 2–5, reflecting moderate enrichment for MOJ, RAD, LIT, VRH, CAT, ZAG, SRM, and BEO (Table 3). However, the percentage of bioavailable Zn was below 4% and $E_i < 40$, indicating an insignificant ecological risk (Tables 2 and 4).

This study revealed that PTE levels in riparian soil increased with distance from the Sava's source; however, for the most part, their levels in low-water conditions do not pose a potential risk to the riverine environment, the only exception being the high total Cd concentrations in soil in the lower reaches of the Sava. The remobilization of this element under extreme flow events from riparian soil into the water column could pose a considerable ecological risk to the riverine ecosystem.

5 Conclusions

In this study, results obtained from 12 sampling sites revealed that PTE in the upper part of the Sava River was mostly in the range of the threshold values proposed for European soils, with

the exception of Cu, which exceeded the proposed values at CAT, with the impact transferred downstream to ZAG and JAS. Despite the high EF at CAT, though, this element, which is of predominantly anthropogenic origin, is characterized by low individual and overall ecological indices, meaning it does not represent a potential ecological risk in this area. At the downstream sites, there was a noticeable increase in PTE content in the examined soils, with Cd, Cr, Ni, and Zn exceeding the proposed values. This indicates that contamination in the riparian zone in comparison to that in previous studies conducted in alluvial soils along the Sava River is rising. It was established that the origin of Cr and Ni in soil is lithological. Due to their low enrichment and bioavailability, Cr and Ni do not present an ecological risk. The other elements were shown to be of both lithological and anthropogenic in origin, depending on the sampling site. The ecological risk indicators for As, Pb, and Zn showed that these elements have a minor to moderate impact. However, the high total Cd content in the lower stretch affected individual and overall ecological risk indicators, demonstrating that Cd could represent a considerable ecological risk for the downstream riparian zone, especially at BEO.

Since there is an increased accumulation of PTE in the lower part of the river, any future flood or high-water events could lead to their remobilization due to a change in physical and chemical soil conditions, posing a considerable ecological risk to the riparian zone.

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