

Water Quality Examination in the Stream Sediment of River Sitnica – the Assessment of Toxic Trace Elements

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ABSTRACT

It is presently well-recognized that the contamination of aquatic sediments in Kosovo is a widespread phenomenon. The observation of the chemical analyses of water and sediments is essential to determine the extent of trace elements in these ecosystems. Hence, in order to be able to determine the mass concentration of the predominant elements present in river Sitnica, inductively coupled plasma mass spectroscopy (ICP-MS) was used for this research. Moreover, a comparison of the mass concentration of water elements with the mass concentration of the elements found in the Sitnica sediments was provided. Utilizing the results obtained from the ICP-MS analysis, it was shown that the mass concentration of nickel, arsenic, zinc, cadmium, lead, copper, aluminum in certain water sample stations exceeds the permissible sediment quality criteria. Considering the toxicity that heavy metals exert on the aquatic environment, this research aimed to assess and measure the metal toxicity, because only then, a proper solution can be suggested to sustain freshwater ecosystems and to prevent constant damage from the human activity.

Keywords: Sitnica, river water, stream sediments, quality assessment, inductively coupled plasma mass spectroscopy.

INTRODUCTION

Water is essential for all processes associated with life. Among them, riverine systems are the most important freshwater resource for humans. These complex ecosystems are continuously being damaged and their quality is currently threatened by a combination of human activities as are overexploitation, as well as microbiological and chemical contamination. Even though water constitutes the majority of the Earth's surface, only 0.3% of it is available for human use. Because of the continuously increasing population, water sources are gradually becoming scarce. Besides, what further adds to this phenomenon is the mixture of several contaminants that are formed through the soil and rock weathering and natural processes, as well as anthropogenic activities

which include industrial processes, agricultural activities, and population growth (Bhat et al. 2019). According to (Ruminaite 2011), the anthropogenic activity harms the environment and it is considered the main cause of water pollution and stream sediments. Not only have heavy metals become a worldwide problem, but their feature of being indestructible and their negative effect on organisms has been widely recognized during the last decades. This is because they affect the marine systems as well. Rivers transport heavy metals and transfer them to the coastal marine.

Furthermore, what further adds to water pollution is the excessive use of nature and the unrestrained use of natural resources by the human activity. Among these activities, the inappropriate processing of industrial waste is considered as the main cause of the contamination of the

world water resources. This contamination consists mainly of toxic metals (mercury, lead, cadmium, copper, zinc, nickel, etc). During the past few years, significant discoveries and progress have been made in the field of water toxicology, mainly in the distribution of heavy metals and their effect on the aquatic ecosystems. The main challenge for scientists nowadays is to determine the distribution of the trace elements and investigate all their physical and chemical properties in natural water sediments-systems, because they are continuously changing in their values (Arab and Alshikh, 2010; Eric and Charlotte, 1999). By having the exact results of these elements in the Sitnica River, protection and other detoxification methods can be proposed and applied for the affected locations.

Even though the ecosystem is equipped with the best mechanisms for “auto purification”, it is worth mentioning the processes that regulate the normal value of water and its systems, such as precipitation, adsorption, dissolution, and redox processes. Among the elements that cause the greatest toxicity for the flora and fauna are copper, cadmium, lead, and zinc, usually in high concentration, which through their connection with natural ligands minimizes their physiologic activity (Faiku et al. 2011; Troni et al. 2012). Besides, heavy metals are toxic for the human health as well. The river water, which consists of high concentration of heavy metals, is mostly used for the irrigation purposes. People use river sources for irrigating their agricultural lands. Thus, as we eat the products grown under such circumstances, it is likely that we carry the same metals in our organisms.

On the basis of the previous studies, it can be concluded that the rivers in Kosovo are severely damaged or in some cases even destroyed. Taking into consideration the increasing population growth and overexploitation, this research aimed to provide a systematic research of the water resources of the Republic of Kosovo especially the Sitnica River to further present an essential pattern that supports healthy river ecosystems and better management of water resource.

Through this study, the researcher aimed at proposing a network which can be used to permanently monitor stations throughout the whole river bank. By utilizing the Water Framework Directive and samples of this study, the authorities and Kosovar citizens would be able to use the same template for more advanced studies in the River Sitnica.

EXPERIMENTAL PROCEDURE

Research setting

Sitnica River is the longest river that flows in the Republic of Kosovo and along with its flow, it accumulates a great number of streams and wastewater from different urban centers and factory districts. Apart from being the longest, Sitnica River is also among the most polluted ones in the Republic of Kosovo. What heavily impacts the water chemistry and the appearance of trace elements is the hydrographic basin and its geological features. This can be due to three divisions of rocks that are present within the territory of the Republic of Kosovo, namely magnetic, sedimentary, and metamorphic which belong to the Precambrian to Quaternary ages. The sediments of the rivers in Kosovo consist of gravel materials which are mainly not consolidated or poorly consolidated sand.

For the above mentioned facts, the Sitnica River was chosen for this research. Consequently, the sampling sites of the river Sitnica were geographically chosen by using a computer system (GIS - geographic information system). In these water settlements, over a while, the researcher systematically observed and monitored the values of the toxic and other elements found in the Sitnica River. In order to come to a conclusion, the instrument used in this research was previously tested for its reliability and validity. The analysis of the results was done using the inductively coupled plasma mass spectroscopy (ICP-MS). As a consequence, the fundamental elements that were found prevalent in this research were further highlighted.

METHODOLOGY

Under the methodology section, the experimental procedure is as follows. Initially, the plastic bottles that were used for the research were cleansed with sampled water more than three times. Afterwards, a label with the date and the name of the source of samples was placed above each bottle. Subsequently, the samples were placed into a refrigerator (4°C) to be further analyzed within the chemical laboratories. They were tested and pretested so that the average values would be reliable for the research. The researcher has carefully chosen the specific locations where he assumed that high rates of toxicity would be

expected. This is because of the proximity of factories, industrial areas, settlements, etc.

The researcher administered the experimental procedures of sampling, preservation, and analysis according to the standard methods of water examination (Sikošek 1971; Ivković et al. 1983; Hurd et al. 1991; Skoog et al. 1992; APHA, 1998; Baba et al. 1998). Besides, a manual collection method was used to sample the stream sediments that used to flow along with running water. The researcher used a plastic pail with dimension 25×8×3 cm. In order to avoid probable contamination of the river banks, the sampling was carried out at a sufficient distance.

Moreover, in each sample settling the exact needed material for geochemical analysis was provided (1–2 kg sediment for fraction <63 µm). The material was transferred into plastic containers. Over three weeks, the samples were stored at room temperature 20–25° C for the purpose of drying. Lastly, after this procedure, coarse and sand material was divided using a sieve of 40 meshes and afterwards a standard sieve of 63 µm, Fritsch (Germany). Following the requirements, after the treatment, the samples were kept in the refrigerator. As for the chosen geographical positions, the model “GEKO, GARMIN”, 12 channels was used.

The study area with the sampling locations and the specific details about all sampling sites are elaborated in Table 1.

Instrument

In order to compare and contrast the results, Statistica 6.0 (2001) software package was

applied. The fundamental reason for using this program is because it helped the researcher mainly with the statistical calculations of the results such as descriptive statistics, and frequency histograms. Apart from that, a two-dimensional box plot diagram was utilized to define the irregularities (extremes and outliers) for data solutions.

RESULTS AND DISCUSSIONS

Predominant trace elements according to ICP-MS analysis

In tables 2 and 3, the concentration of chemical parameters in the water sample and in-stream sediments is shown (fraction <63 µm). For chemical assessment and statistical calculations of the water quality of Sitnica River, a database with tables and figures is given ().

Statistical analysis

A series of a visual representation is used for statistical analysis. Hence, the fundamental parameters in eight different samples of water and stream sediments (fraction <63 µm) are presented in the Figures 2, 3 and 4. The evaluation conducted for this research can serve as a statistical parameter until a large scale of data collection is gathered. In order to provide valuable insight, the calculation was performed for each element through arithmetic and geometric means. Likewise, the minimal, medial and maximal mass concentration of the element, their variables and standard deviation is clearly expressed as well.

Table 1. A thorough description for each Sampling Station

Sample	Location	Coordinates	Sources which caused pollution
S ₁	Rubovc	42°12'59" N 21°15'12" E	The settlement, agriculture land.
S ₂	The road cross of Lipjan city	42°12'59" N 21°15'12" E	Wastewater, agriculture land
S ₃	Lismir village	42°12'59" N 21°15'12" E	Road, wastewater
S ₄	Palaj village	42°12'59" N 21°15'12" E	Road, wastewater from Palaj village
S ₅	Plemetin village	42°12'59" N 21°15'12" E	Agriculture, road, wastewater from Plemetin village
S ₆	The bridge of Pestova village	42°12'59" N 21°15'12" E	Road, settlement, wastewater
S ₇	Vushtria city	42°12'59" N 21°15'12" E	The settlement, wastewater.
S ₈	North Mitrovica city	42°12'59" N 21°15'12" E	Road, settlement

Table 2. The mass concentration assessment of 30 elements determined in the water of Sitnica River.

Element μgdm^{-3}	Method/Detection Limit	S ₁	S ₂	S ₃	S ₄	S ₅	S ₆	S ₇	S ₈
Li	ICP-MS/1	4	14	13	7	5	16	12	9
Mg	ICP-MS/1	5200	1950	>2000	17200	21000	1950	16500	19200
Al	ICP-MS/2	65	44	145	14	16	44	92	18
Si	ICP-MS/200	4400	7900	5900	7500	7500	9500	9200	8700
K	ICP-MS/30	1200	2810	3540	6340	5410	7500	8200	6900
Ti	ICP-MS/0.1	1.2	1.2	2.4	0.9	1.1	3.4	1.3	1.4
Va	ICP-MS/0.1	0.2	0.5	1.20	1.20	1.30	0.9	1.6	2.1
Cr	ICP-MS/0.5	0.7	0.8	1.20	0.70	0.80	0.80	1.60	1.5
Fe	ICP-MS/10	80	120	150	140	150	170	180	120
Co	ICPMS/0.005	0.245	0.147	0.698	0.428	0.208	0.412	0.525	0.320
Cu	ICP-MS/0.2	1.20	1.10	4.70	1.70	1.50	2.50	3.50	6.70
Ge	ICP-MS/0.01	0.01	0.01	0.02	0.01	0.03	0.01	0.02	0.04
As	ICP-MS/0.3	0.85	4.10	4.65	3.96	2.68	4.15	5.85	4.87
Se	ICP-MS/0.2	0.2	0.2	0.6	0.5	0.8	0.7	0.6	0.5
Br	ICP-MS/1	51	33	79	74	85	45	88	92
Rb	ICP-MS/0.005	0.778	0.956	1.92	1.54	2.20	1.22	1.97	2.46
Sr	ICP-MS/0.04	26.3	25.2	32.2	56.0	54.9	56.2	67.4	64.5
Zn	ICP-MS/0.5	95.5	98.1	77.1	77.1	152.2	125.2	133.4	129.4
Mo	ICP-MS/0.1	0.1	0.3	0.2	0.3	0.3	0.2	0.2	0.3
Cd	ICP-MS/0.01	0.01	0.02	0.03	0.02	0.03	0.04	0.24	0.42
Sb	ICP-MS/0.01	0.16	0.12	0.21	0.25	0.36	0.34	0.32	0.36
I	ICP-MS/0.01	2	2	4	2	3	1	3	3
Cs	ICP-MS/0.001	0.114	0.235	0.171	0.111	0.25	0.171	0.213	0.215
Ba	ICP-MS/0.1	15.2	35.1	44.1	2.8	2.3	43.1	32.9	32.3
La	ICP-MS/0.001	0.279	0.140	0.320	0.311	0.232	0.331	0.222	0.255
Ce	ICP-MS/0.001	0.360	0.206	0.532	0.142	0.203	0.255	0.245	0.141
Tl	ICP-MS/0.001	0.003	0.004	0.003	0.002	0.002	0.002	0.003	0.003
Pb	ICP-MS/0.01	0.75	0.62	2.35	1.22	0.87	1.75	1.32	1.65
Bi	ICP-MS/0.3	<0.3	<0.3	<0.3	<0.3	<0.3	<0.3	<0.3	<0.3
U	ICP-MS/0.001	0.113	0.609	0.606	0.752	0.601	0.501	0.607	0.705

Discussion of ICP-MS analysis of water samples

Among the main elements obtained by ICP-MS, a graphical representation of data was provided for zinc, antimony, lead, arsenic, copper, cobalt, chromium, cadmium, barium, and uranium (see Table 2). It is worth emphasizing that the anomalous values (extremes and outliers) are presented using a boxplot approach (Tukay, 1997) for all sample's stations. A comparison was conducted between the toxic elements found in this research (zinc, antimony, lead, arsenic, copper, cobalt, chromium, cadmium, barium, and uranium) with current criterion set by SMSP regarding the sediment quality Falconbridge NC, SAS (2005).

The mass concentration of aluminum was prevalent in the sample location S₃ (145 μgdm^{-3}) whereas the mass concentration of iron seems to be dominant in the water sample location S₆ (180 μgdm^{-3}), and this may be a direct impact from the geological constitution of rocks: alluvium gravel, sand silt, limestone, marlstone, calcarenite, olistoliths of cherty limestone and silty deposits. The mass concentration of aluminum above 200 mgdm^{-3} causes significant toxic effects. The mass concentration of chromium is between 0.6 μgdm^{-3} (the lowest level in S₂) to 1.6 μgdm^{-3} (the highest level in sampling station S₆). The mass concentration of arsenic is between 0.85 μgdm^{-3} (the lowest level in S₂) to 5.85 μgdm^{-3} (the highest level in sampling station S₃ and S₇, respectively),

Table 3. The distribution of 51 chemical substances determined in stream sediments.

Element /ppm/	Method /Detection Limit	S ₁	S ₂	S ₃	S ₄	S ₅	S ₆	S ₇	S ₈
Eu	ICP-MS/0.05	0.76	0.85	1.44	1	0.78	0.98	1.16	1.48
Tb	ICP-MS/0.1	0.6	0.60	0.9	0.7	0.8	0.7	0.9	1.2
Dy	ICP-MS/0.1	3.4	3.0	5.8	4.8	4.2	4.9	3.8	5.4
Ho	ICP-MS/0.1	0.6	0.8	1.2	0.9	0.8	0.65	0.80	1.3
Er	ICP-MS/0.1	1.8	1.8	2.8	2.4	1.8	1.9	2.5	3.9
Tm	ICP-MS/0.05	0.20	0.20	0.46	0.35	0.35	0.55	0.55	0.97
Yb	ICP-MS/0.1	1.7	2.7	2.7	2.5	2.8	3.1	2.4	2.1
Lu	ICP-MS/0.04	0.35	0.37	0.44	0.53	0.55	0.47	0.54	0.87
Ta	ICP-MS/0.1	0.5	0.6	0.9	0.9	0.9	1.2	1.7	1.9
Ti	ICP-MS/0.1	0.3	0.3	0.5	0.6	0.6	0.7	0.9	0.9
Bi	ICP-MS/0.4	0.5	0.5	0.4	0.6	0.9	1.8	1.4	1.9
Th	ICP-MS/0.1	6.4	5.9	8.3	7.5	7.7	9.5	8.2	8.2
U	ICP-MS/0.1	1.4	1.5	2	1.6	1.5	1.5	1.7	1.8
Rb	ICP-MS/2	55	67	65	51	53	57	74	85
Sr	ICP-MS/2	65	40	70	45	59	92	85	125
Y	ICP-MS/0.1	20	19	33	27	18	21	25	28
Zr	ICP-MS/4	140	130	290	268	230	295	440	354
Nb	ICP-MS/1	6	6	12	9	16	8	12	15
Sn	ICP-MS/1	4	3	35	33	5	27	35	32
Sb	ICP-MS/0.5	1.4	1.2	1.6	0.8	1.5	1.4	1.3	1.4
Cs	ICP-MS/0.5	3.15	2.77	3.50	2.25	2.50	2.45	3.15	3.88
Ba	ICP-MS/3	218	285	340	245	220	240	275	375
La	ICP-MS/0.1	18.0	17.80	32.1	21.4	20.1	20.9	22.6	29.2
Ce	ICP-MS/0.1	37.8	37.9	65.4	56.8	45.5	65.2	75.2	75.9
Pr	ICP-MS/0.05	4.51	4.52	8.5	8.31	8.95	9.15	10.12	11.90
Nd	ICP-MS/0.1	19.8	19.5	33.4	31.8	35.6	32.6	35.0	44.2
Sm	ICP-MS/0.1	3.8	3.6	6	4.5	3.2	6.6	8.5	8.9
Be	ICP-MS/1	1	1	2	2	3	1	1	2
V	ICP-MS/5	85	80	86	83	88	92	102	115
Co	ICP-MS/1	20	15	18	18	19	18	25	53
Ni	TDICP-MS/1	126	116	125	132	160	185	195	195
Cu	TDICP-MS/1	30	38	58	37	56	67	76	82
Zn	TDICP-MS/1	88	91	115	86	127	106	184	195
S	TDICP-MS/0.01	0.030	0.063	0.044	0.020	0.090	0.055	0.055	0.081
Ag	TDICP-MS/0.3	0.4	0.4	0.4	0.3	0.6	0.8	0.9	2.1
Pb	TDICP-MS/5	21	25	26	21	37	44	68	68
Ga	FUS-MS/1	12	11	12	10	14	15	12	18
Ge	FUS-MS/0.04	2	1	2	1	1	3	2	3
Au	INAA/2	14	2	42	2	42	47	44	35
As	INAA/5	27.4	18.5	20.8	17.1	20.7	18.6	19.2	15.4
Br	INAA/0.5	6.15	3.17	10.18	0.5	7.21	17.35	12.5	15.2
Cr	INAA/5	350	320	384	725	613	450	650	552
Ir	INAA/5	<5	<5	<5	<5	<5	<5	<5	<5
Sb	INAA/0.2	1.4	1.8	1.7	2	2.5	2.8	4.4	2.5
Sc	INAA/0.1	12.0	12.6	13.2	12.5	16.2	15.3	17.7	16.9

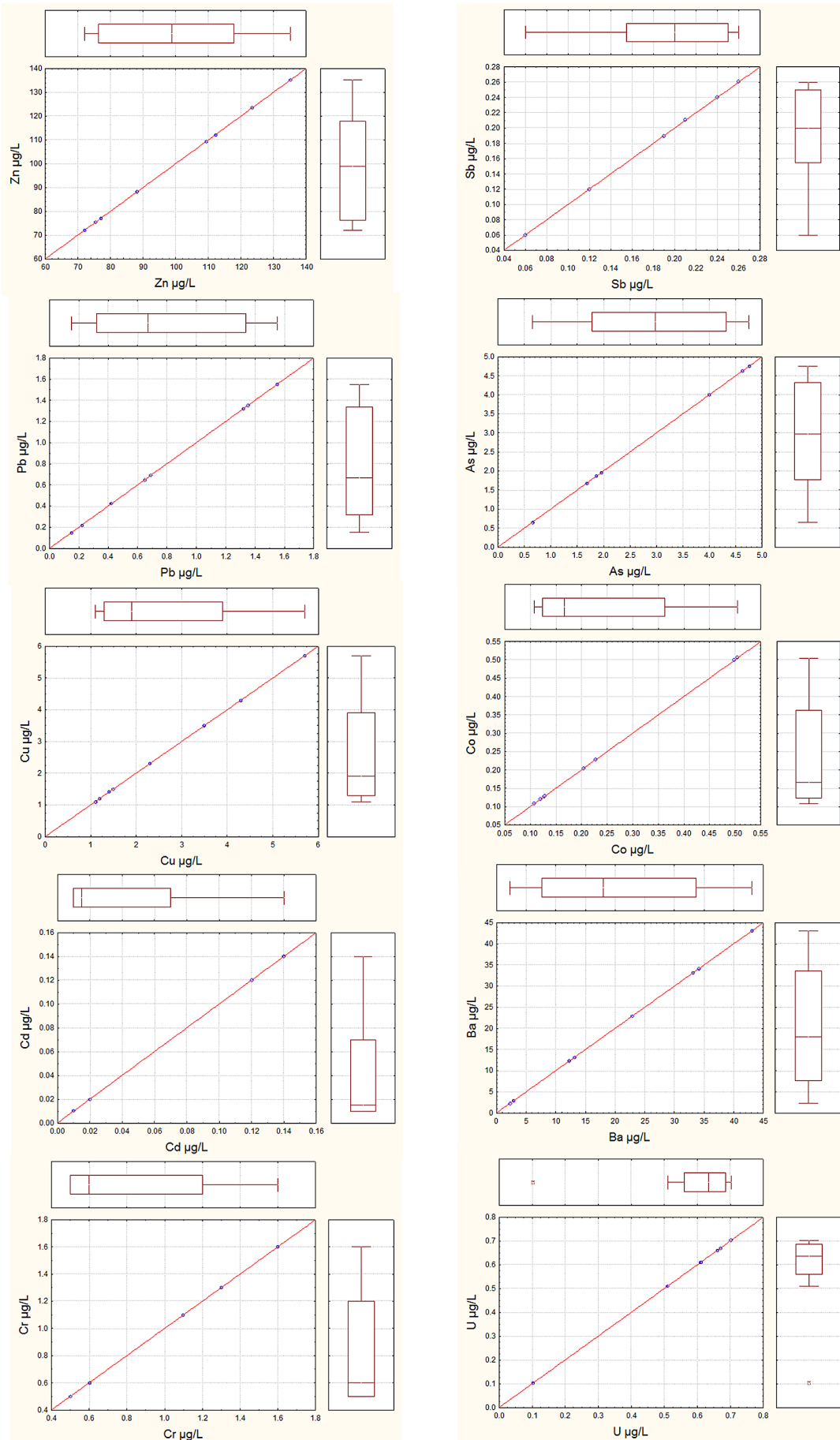


Figure 1. Scatter plot with box plots diagrams of some measured variables in water samples.

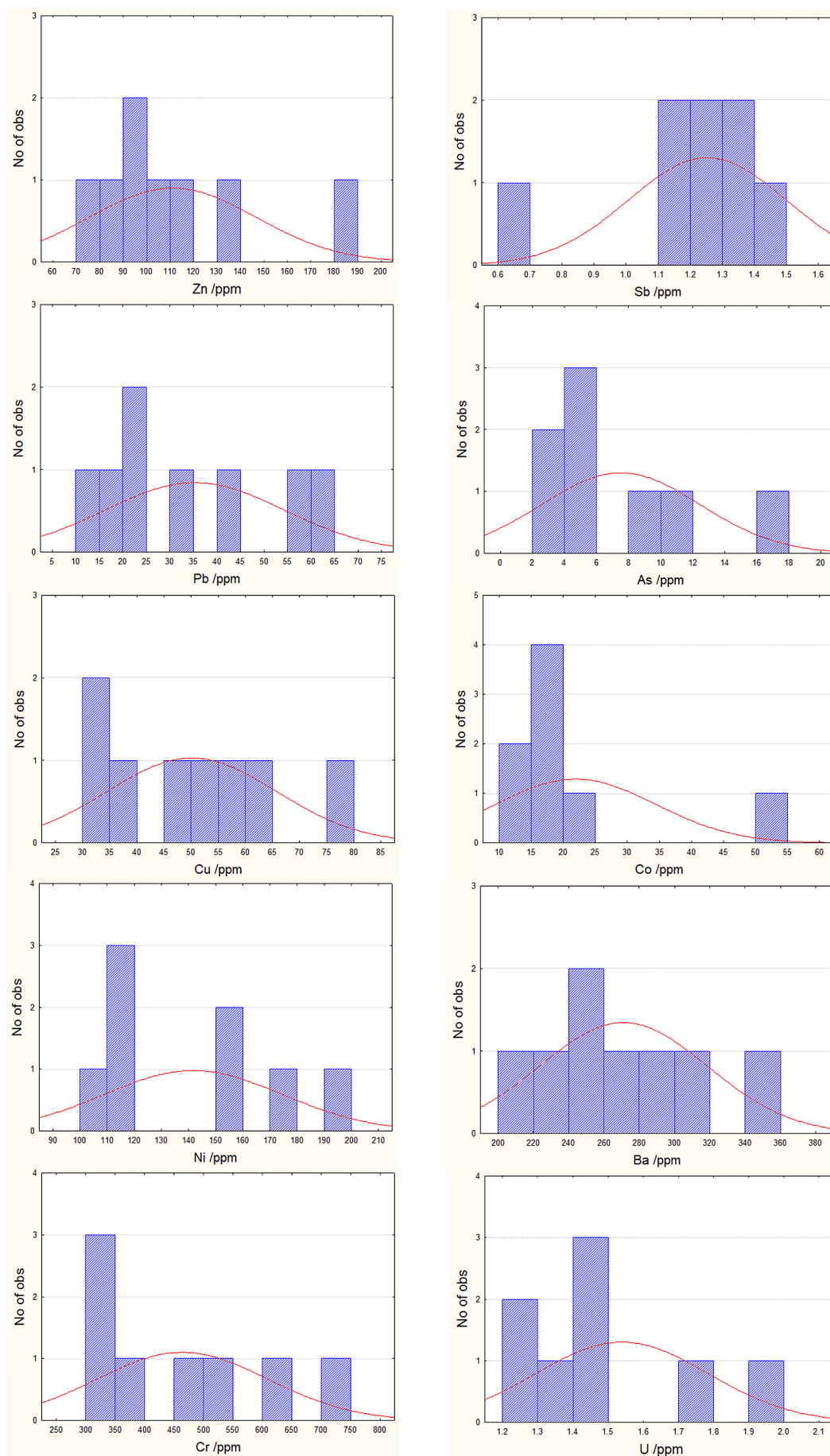


Figure 2. Frequency histograms of 10 measured variables in sediment samples.

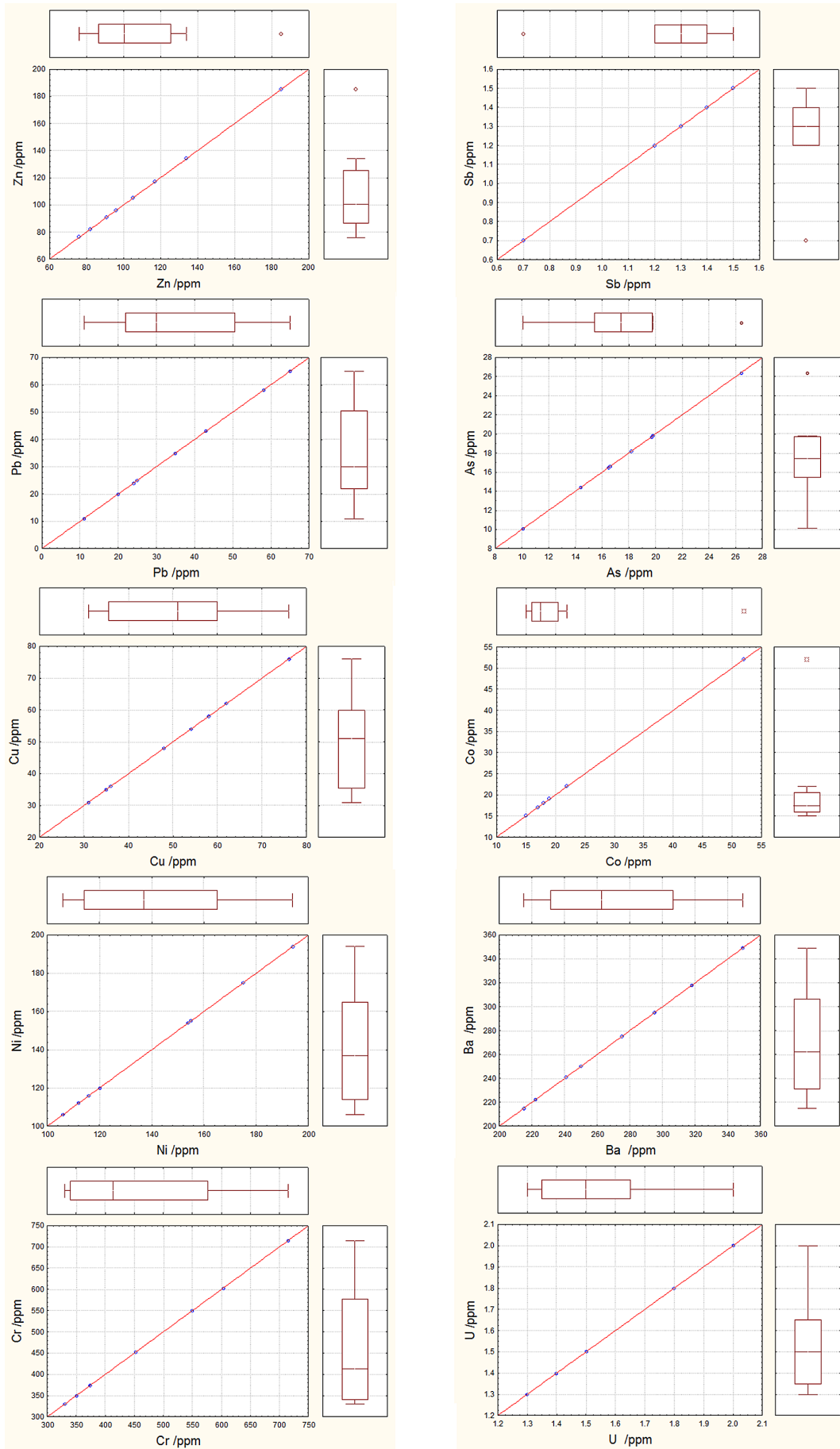


Figure 3. Scatter plot diagrams of some measured variables in sediment samples.

with a standard deviation of 1.573. The mass concentration of cobalt is between $0.698 \mu\text{gdm}^{-3}$ (the lowest level in S_5) and $0.525 \mu\text{gdm}^{-3}$ (the highest level in sampling station S_7). The mass concentration of copper is from $1.10 \mu\text{gdm}^{-3}$ (the lowest level in S_2) to $6.70 \mu\text{gdm}^{-3}$ (the highest level in sampling station S_8). The mass concentration of selenium is between $0.2 \mu\text{gdm}^{-3}$ (the lowest level in S_1 , S_2 , and S_6) to $0.60 \mu\text{gdm}^{-3}$ (the highest level in sampling station S_7), (with standard deviation 0.151). All these ecotoxic elements chromium, cobalt, copper, and selenium which cause significant toxic effect, are below the allowed concentration $50 \mu\text{gdm}^{-3}$ WHO (2004–2007). Antimony, barium, lead, thallium, zinc, and uranium mass concentrations in all water samples were under the WHO-allowed values. Another element that was prevalent in the river water regarding their mass concentration is the cadmium. The concentration of cadmium is under $0.2 \mu\text{gdm}^{-3}$ according to allowed values.

Discussion of ICP-MS analysis of sediment samples

As for the analysis of sediment samples, nickel was predominant in all sediment samples varying from 116 to 195 ppm. The mass concentration of nickel exceeds the permissible concentration in all sediment samples. The concentrations of Ni over 75 ppm, as shown in our results, causes high toxicity in the riverine system. Copper was also highly widespread in all sediment samples. The concentrations of Cu in samples S_5 - S_8 were above 50 ppm, which causes significant toxic effects.

The chromium concentrations in all eight sediment samples exceeded the allowed concentrations for sediment (the allowed concentration of chromium is under 110 ppm). The cobalt concentrations only in sediment sample S_8 (53 ppm) exceeded the allowed concentrations for sediments (the allowed concentration of cobalt is under 50 ppm). Similar silver concentrations in sampling stations S_5 - S_8 exceeded the allowed values (allowed concentrations are under 110 ppm). Antimony also appeared to be concentrated in the river sediments but significantly under the allowed values from 500 ppm. The concentration of Zn, As and Pb were found low, and following the Canadian standards for sediments, they were under allowed concentration. These exceeded the mass concentration of some elements in several sampling stations that are directly impacted by the

geological constitution of rocks: alluvium gravel, sand silt, limestone, marlstone, calcarenite, olistoliths of cherty limestone, and silt deposits. Again, the data collected for this research, obtained from ICP-MS analysis reveals the anomalous variables for zinc, antimony, lead, arsenic, copper, cobalt, chromium, cadmium, barium, and uranium) for the whole region (Tukay, 1977).

The results of the box plot detection, show several contaminated areas in stream sediment of the river of Sitnica (sample stations S_1 and S_8). The outlier values of arsenic and zinc were registered in sample S_1 and S_8 , respectively and the extreme value of cobalt (52 ppm) was registered only in sample station S_8 . It is suggested that the geological origin has influenced the appearance of some of the elements. For instance, zinc and arsenic are the elements that were found in this research that derive from the Late Paleozoic rocks. Besides, the auto-purification mechanism was introduced in the example of arsenic, which appeared to be in a dense concentration in water at station six S_6 .

CONCLUSIONS

Potable water has a significant impact on the water industry in the Republic of Kosovo and its excessive contamination has gradually led to total contamination. This contamination is a prevailing phenomenon and deserves considerable attention because of the toxic effect it has on human health. Besides, we should not overlook the economic damage caused by this contamination. Despite the ongoing effort for the aquatic environment, only a few analyses were carried out over the last years. Considering this fact, the study was carried out and as a conclusion, it emphasizes that the mass concentration of aluminum in certain water stations is dominant and that the value above $200 \text{mg}\cdot\text{dm}^{-3}$ causes significant toxic effects. All other elements in the water of the Sitnica River were found as less dominant regarding their mass percent concentration compared to other elements and were under the permissible concentration of WHO (do not cause significant toxic effects). The mass percent concentration of cobalt, copper, nickel, chromium, and silver in river water stations exceeds the permissible criterion and that value causes significant toxic effects. All other elements in the water of the Sitnica river were found in low concentrations and were under the

current criterion set by SMSP for sediment quality, Falconbridge NC, SAS (2005). The results of the box plot detection indicate several contaminated areas in the stream sediment of Sitnica river. The outlier variables of arsenic and zinc were registered in two samples and the extreme value of cobalt was registered only in a sample station.

These exceeded mass concentrations of some elements in water and sediment sampling station are directly impacted by the geological composition of rocks: alluvium gravel, sand silt, limestone, marlstone, calcarenite, olistoliths of cherty limestone and silt deposits. Possible remediation at critical locations from Sitnica River, flowing towards Kosovo, would be desirable. Taking into consideration, in general, that the rivers in our country are still not under the permanent control of the government and the laboratories are still not equipped with adequate gadgets, there are still communities and particularly the academic staff of the Faculty of Chemistry willing to volunteer and participate in the awareness activities regarding the importance of riverine systems.

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