

Study on the quality of ground, spring and river waters in South–East Serbia

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Abstract

The study deals with mineral characterisation of natural waters from South–East Serbia. The contents of aluminium, arsenic, calcium, cadmium, cobalt, chromium, copper, iron, potassium, magnesium, manganese, sodium, nickel, lead and zinc were analysed in spring, ground and river waters by inductively coupled plasma-atomic emission spectrometry (ICP-AES) technique. The study area was in the Southern Serbia, and included slopes of Rtanj, Ozren, Bukovik, Vrdnik and Čemernik mountains, and the valley of South Morava. Obtained contents were compared with Serbian regulations on the quality of water for human use, and directive of World Health Organization (WHO) for maximum allowed concentrations of chemical substances. High contents of macro-elements, namely calcium, magnesium and potassium, were detected in several spring and ground water samples which are believed to be due to direct influence of rock minerals. Some water samples contained iron, manganese and copper in concentration up to 168.3, 8.10 and 14.9 µg dm⁻³, respectively, but within the permissible limits. Other heavy metals were not detected in analysed samples. Based on the derived results, tested ground and spring water samples have significant potential to be used as sources for the production of bottled water, but further investigations are necessary. Additional investigations have to be focused on complete physical, chemical and microbiological assessments of water resources. Systematic hydrogeological assessment also should be performed in all seasons. In the meantime, precautionary measures should be immediately taken to protect and preserve these water resources.

Keywords: Water quality, minerals, heavy metals, water resources, water management.

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Water is the most precious and essential natural resource. Out of all the water on Earth, only small fraction (about 2.5%) of it is fresh, non-saline water, suitable for human consumption [1]. Non-saline water is generally obtained from two principal natural sources: surface waters, such as lakes, rivers and streams, and ground waters, such as spring and well waters. Nowadays water resources available for human consumption are being subjected to continuous environmental stress and pollution. The pollution of water systems affects humans and the natural environment worldwide. Among wide diversity of contaminants affecting water resources, heavy metals receive particular concern considering their strong toxicity, even at low concentrations due to their accumulative effects [2]. Heavy metals exist in natural waters in colloidal, particulate and dissolved forms [3]. Although trace

amount of some metals are essential for humans, any excessive exposure to heavy metals can result in adverse manifestations. The occurrence of heavy metals in ground and surface waters can be due to natural sources, such as dissolution of naturally occurring minerals containing trace elements in the soil zone. Human activities and other anthropogenic sources such as mining, fuels, smelting of ores and improper disposal of industrial wastes, significantly contribute to elevated levels of heavy metals in waters. Surface water bodies are particularly vulnerable to contamination from industrial and municipal wastewater, leaching or runoff of agrochemicals and dissolution of air-borne pollutants [4]. In ground waters greater part of the soluble constituents comes from soluble minerals in soils and sedimentary rocks and a much smaller part has its origin in the atmosphere and surface water bodies.

In Serbia there are two regulations concerning the quality of water for human use: Regulation on the hygienic acceptability of potable water [5,6], and Regulation on quality and other requirements for natural mineral water, spring water and bottled drinking water [7]. The Regulation on the hygienic acceptability of

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potable water defines the maximum allowed concentrations (MAC) of chemical substances in water for public water supplies. Regulation on quality and other requirements for natural mineral water, spring water and bottled drinking water defines the MAC of certain chemical parameters that can be a risk to human health, indicators of water quality and nomenclature of mineral waters. Guideline values have been determined for those chemical components that are considered to have significant potential to harm human health at concentrations above the specified limits. Guideline values should not be exceeded in public water supplies. It should also be noted that in some cases exceeding the guideline values may not always be a matter for immediate concern, but rather a trigger for a follow-up action.

Water analysis plays a decisive role in the regulations that determine water quality for specific uses, especially for drinking water quality [8]. Due to the importance of drinking water for human life, lack of water sources and the increased consumption of mineral waters, there is a need to assess the quality of water from various potential water resources. Besides this, as a part of water resources management, water protection measures need to be prioritised and implemented in order to ensure sustainability for future generations.

Serbia has significant surface water and ground water resources, sufficient to fulfil its current economic and social requirements. According to the density of water resources and the diversity of physicochemical properties of mineral waters, the territory of Serbia belongs to the one of the most resourceful areas of the European continent, but only a small quantity of these mineral waters is used for bottling [9]. In 2008 there were 30 bottling mineral water plants in Serbia [9], however, due to large number of unexamined water sources, it is believed that the potential is much higher.

The objective of this study was to evaluate the quality of spring and ground waters of the area on the slopes of Rtanj, Ozren, Bukovik, Vrdenik and Čemernik mountains and in the valley of river South Morava, in Southern Serbia. The springs of the mountains Rtanj, Ozren, Bukovik, Vrdenik and Čemernik are known for their significant amounts of water relative to Serbia and generally good quality of underground and spring waters. The examined region located at South of the Balkan Peninsula is not fully occupied by industries yet, ensuring somewhat a good quality. Water samples from river South Morava were also subject of this analysis, since some examined wells were situated close to this river. The content of aluminium, arsenic, calcium, cadmium, cobalt, chromium, copper, iron, potassium, magnesium, manganese, sodium, nickel, lead and zinc were analysed in spring water, ground and river water. Emphasis was placed on the contents of some heavy

metals such as lead, copper and cadmium, which are very toxic, even at low concentrations due to the accumulative effects. The measurements were conducted using inductively coupled plasma-atomic emission spectrometry (ICP-AES) technique.

EXPERIMENTAL

The study area

The study area is located in the South-Eastern Serbia, and includes the slopes of Rtanj, Ozren, Bukovik, Vrdenik and Čemernik mountains and the valley of South Morava. The study region lies between the longitude 21.555 to 22.32882 and latitude 42.02009 to 43.79576. The physicogeographical position of the basin is specific due to its geographic affiliation to the mountainous Carpathian-Balkan (middle and east part) and Peripannonian (western part) regions in Serbia. The region included the territory between the valley of the South Morava down river flow in the west, Balkan mountain range in the east, the mountains Bukovik and Rtanj in the north, and the mountains Vrdenik and Čemernik in the south. Investigated area is shown in Figure 1.

Sample collection and treatment

Water samples were collected applying random and haphazard sampling from 19 different sampling sites in South-Eastern Serbia region (Figure 1). Sampling sites are described in Table 1.

Collected samples included spring, ground and river waters. River water samples were collected randomly, while ground water samples were collected by applying haphazard sampling approach [10]. Spring water samples were collected by applying a combined random and haphazard sampling approach. All samples were collected in April 2013. The river and spring water samples were collected by immersion of plastic bottles below the water surface. Groundwater samples were directly collected from wells. Before sampling, all plastic bottles were rinsed with 5% HNO₃, and then with ultrapure water. Plastic bottles were rinsed three times with the water sample before sampling. The water samples immediately upon collection were well mixed with 2 cm³ HNO₃ (Suprapur®, Merck, Germany) per litre sample and capped tightly, in order to preserve the samples. Preserved samples were transported in ice-packed coolers to the laboratory for analysis within 24 h. Prior analyses, the samples were filtered through 0.45 µm membrane filters.

Analytical method

For element analysis in collected water samples inductively coupled plasma-atomic emission spectrometry (ICP-AES) was used. Analysis was performed using ICP-

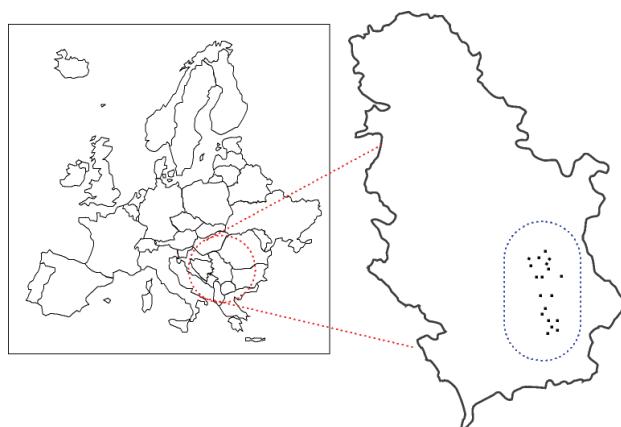


Figure 1. Map of investigated area – location of the sampling sites are marked.

Table 1. Sampling sites for different water samples

Sample number	Origin	Latitude (UTM)	Longitude (UTM)	Description
1	River water	43.561082	21.640528	8 km downstream from Aleksinac
2	River water	43.532823	21.653012	4.8 km downstream from Aleksinac
3	River water	43.527067	21.712049	Aleksinac
4	River water	43.451610	21.746339	14.5 km upstream of Aleksinac
5	River water	43.371209	21.768637	27.4 km upstream of Aleksinac
6	River water	42.834799	22.130302	Predejane
7	River water	42.699359	22.064817	Vladičin Han
8	Spring water	43.772395	21.933947	Eastern slope of Rtanj
9	Spring water	43.795756	21.760225	Northwestern slope of Rtanj, Krivi vir
10	Spring water	43.625265	21.853867	Northeastern slope of Ozren, Gradašnica
11	Spring water	43.551985	21.812711	Southern slope of Ozren, Lipovac
12	Spring water	43.551658	21.810265	Southern slope of Ozren, Lipovac
13	Spring water	43.72211	21.555004	Northwestern slope of Bukovik, Šetka
14	Ground water	42.767962	22.087643	Džep
15	Ground water	42.691647	22.175345	Surdulica
16	Ground water	43.293247	22.006881	Niška banja
17	Ground water	43.560989	21.833879	Southern slope of Ozren, Lipovac
18	Ground water	42.760121	22.178307	Southwestern slope of Čemernik, Mačkatica
19	Ground water	42.742773	22.328821	Vlasina Rid

-OES, Varian Vista Pro-axial. Concentrations of heavy metals and trace elements were determined by US EPA method 200.7 [11]. Samples were analysed directly by pneumatic nebulization without acid digestion. Stock standards were purchased from J.T. Baker, BAKER Instra-Analysed Reagent Grade (J.T. Baker® 5724 Calcium Plasma Standard, J.T. Baker® 5741 Potassium Plasma Standard, J.T. Baker® 5734 Magnesium Plasma Standard, J.T. Baker® 5746 Sodium, Baker Instra-Analyzed® Reagent Grade and J.T. Baker® 6031-01 Trace Metal Standard I Al, As, Cd, Co, Cr,Cu, Fe, Mn, Ni, Pb, Zn).

RESULTS AND DISCUSSION

The results on elements contents in the analysed water samples are given in the Table 2. Comparison of

Regulations in Serbia with directive of World Health Organization [12] for maximum allowed concentrations of chemical substances is also shown in Table 2.

The obtained results indicated that the concentrations of almost all of the analysed elements and heavy metals in water samples were considerably below the maximum allowed concentrations (MAC) stipulated in Official Gazette of the Republic of Serbia and WHO (Figure 2).

The concentrations of calcium in analysed samples were in the range from 31.4 to 180 mg dm⁻³. All samples met the Regulations for calcium for public water use, but not for natural mineral water, spring water and bottled drinking water. Due to high calcium content, only samples 12, 13, 14 and 19 could be clas-

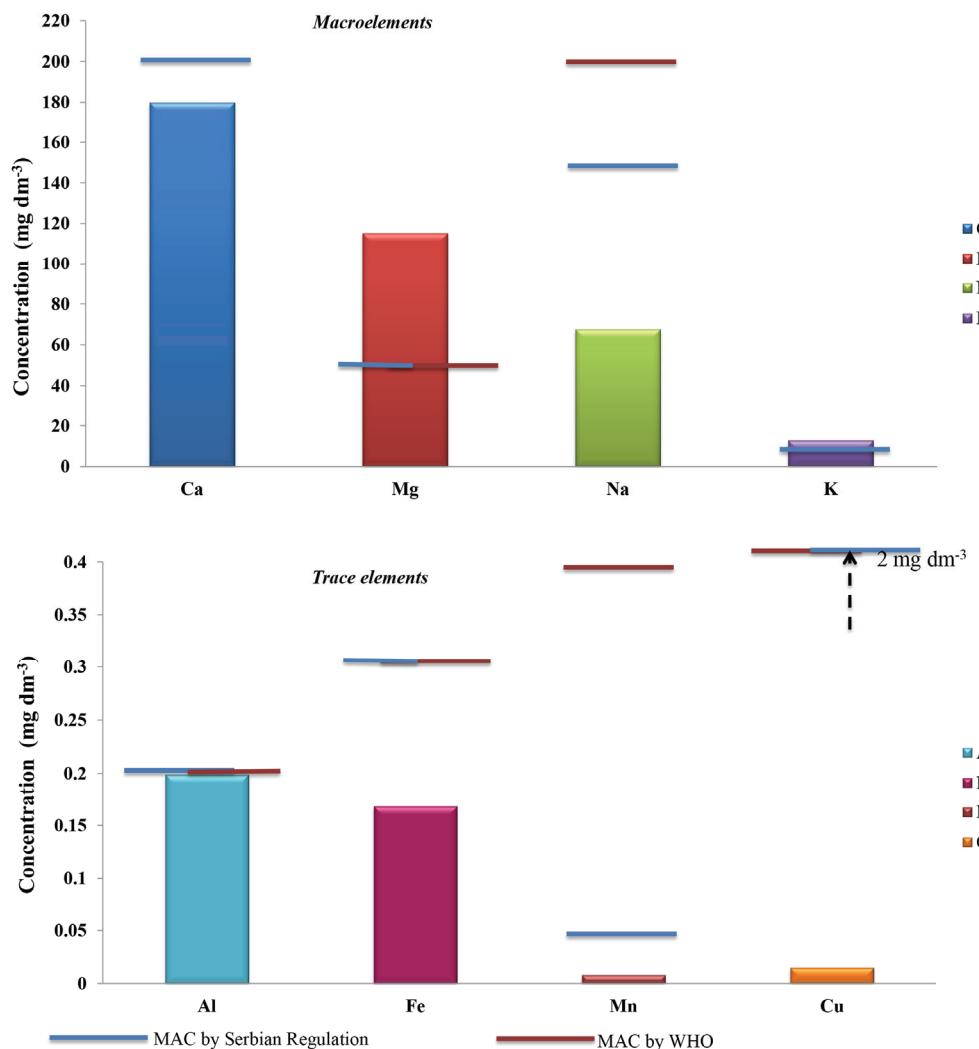


Figure 2. Maximum concentrations of micro, macro and trace elements determined in water samples compared with maximum allowed concentrations.

Table 2. Concentrations of trace and major elements in the analysed water samples (mg dm⁻³); nd – not detected

Sample	Element														
	Ca	Mg	Na	K	Al	As	Fe	Mn	Cd	Co	Cr	Cu	Ni	Zn	Pb
1	85.5	13.28	10.99	2.79	nd ^a	nd	0.013	0.002	nd						
2	93.7	23.45	22.71	4.07	nd	nd	nd	0.003	nd						
3	76.9	17.06	31.24	6.18	nd	nd	0.026	0.005	nd						
4	75.6	12.24	13.57	3.72	0.182	nd	0.004	nd	nd	nd	nd	nd	nd	nd	nd
5	31.4	7.29	18.68	4.42	0.199	nd	0.142	0.006	nd						
6	91.1	25.80	25.47	3.13	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
7	35.8	12.18	13.27	7.73	nd	nd	0.168	0.008	nd						
8	136.0	9.16	4.33	1.41	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
9	112.5	19.60	5.64	2.56	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
10	113.6	19.75	5.88	2.55	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
11	94.6	8.20	3.54	0.69	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
12	150.6	29.31	34.30	1.96	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
13	158.3	73.69	39.58	0.73	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
14	174.9	115.20	67.77	2.18	nd	nd	0.084	0.005	nd						

Table 2. Continued

Sample	Element														
	Ca	Mg	Na	K	Al	As	Fe	Mn	Cd	Co	Cr	Cu	Ni	Zn	Pb
15	125.1	47.27	54.46	0.39	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
16	124.3	37.89	64.17	0.91	nd	nd	nd	nd	nd	nd	nd	0.015	nd	nd	nd
17	148.1	49.53	65.61	2.21	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
18	103.9	51.75	65.41	12.88	nd	nd	0.011	0.003	nd	nd	nd	nd	nd	nd	nd
19	180.0	90.12	38.99	0.75	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
MAC ^a	200	50	150	12	0.2	0.01	0.3	0.05	0.003	–	0.05	0.02	2	3	0.01
MAC ^b	150 ^c	50 ^c	200 ^c	–	0.2	0.01	0.2	0.05	0.003	–	0.05	0.02	2	–	0.01
WHO ^d	–	50	200	–	0.2	0.01	0.3	0.4	0.003	–	0.05	0.07	2	–	0.01

^aMaximum allowed concentrations, Guideline value given by the Regulation on the hygienic acceptability of potable water [5,6], MAC of chemical substances in water for public water supply; ^bmaximum allowed concentrations, Guideline value given by the Regulation on quality and other requirements for natural mineral water, spring water, and bottled water [7]; ^clower limit of nomenclature; ^dGuideline values of WHO [12]

sified as mineral waters in accordance with Serbian Regulation on quality and other requirements for natural mineral water, spring water and bottled drinking water. Magnesium content in examined water samples varied from 7.3 to 115.2 mg dm⁻³. Samples 13, 14, 18 and 19 had content higher of those permitted by Serbian Regulation and by the WHO, however those samples met requirements for natural mineral water, spring water and bottled drinking waters. Drinking water with high levels of calcium and magnesium could provide important health benefits. Magnesium is very helpful in the prevention of some diseases, including hypertension, fluctuation of blood sugar levels, heart arrhythmia, endocrinologic diseases and nervous system diseases. Calcium may be helpful in osteoporosis for many people whose dietary intake is deficient in this nutrient [12]. A review of published data [13] showed that bioavailability of the calcium from waters is comparable to bioavailability of calcium from milk, when studied under similar conditions. Heaney concluded that calcium in high-calcium mineral waters is highly absorbable, noting that high-calcium water functions much like a supplement [13]. Also, waterborne magnesium is more bioavailable than magnesium obtained from foods and water may be a significant source of magnesium [14]. Although studies show that calcium and magnesium are useful minerals in water, there are no guidelines on minimum concentrations of these minerals in drinking water. Contents of calcium and magnesium in water are associated with water hardness. Hard water is not a health risk, but is a nuisance because of mineral build up in plumbings and poor soap and/or detergent performance, if such water is used in household or in industry.

Most water supplies contain less than 20 mg of sodium per litre, but in some countries levels can exceed 250 mg dm⁻³ [15]. Saline intrusion, mineral deposits, seawater spray, sewage effluents, and salt used in road de-icing can all contribute significant

quantities of sodium in water. The relationship between elevated sodium intake and hypertension has been the subject of considerable scientific controversy, but on the basis of existing data, no firm conclusions can be drawn concerning the possible association between sodium in drinking water and the occurrence of hypertension [15]. No health-based guideline value is therefore proposed. However, sodium may affect the taste of a drinking-water at levels above 200 mg dm⁻³ [15]. All analysed samples had satisfactory content of sodium (3.5–67.8 mg dm⁻³).

Potassium is an essential element in humans. It occurs widely in the environment, including all natural waters, in low concentrations. Adverse health effects due to potassium consumption from drinking-water are unlikely to occur in healthy individuals, because it is rapidly excreted in the absence of pre-existing kidney damage. Consequently it is not considered necessary to establish a health-based guideline value for potassium in drinking-water. In analysed samples potassium content was in the range of 0.4–7.7 mg dm⁻³, and the exception was the sample 18, where its content was 12.88 mg dm⁻³. Only this sample had a higher content of potassium than allowed by Serbian regulations for chemical substances in water for public water supply.

Aluminium is one of the most abundant elements found in the Earth's crust. Toxicity of aluminium is associated with continuous low level exposure, which can eventually lead to serious health effects such as Alzheimer's and Parkinson's disease, senility and presenile dementia [2]. Results obtained showed that aluminium was detected in two samples at levels below MAC (0.182–0.199 mg dm⁻³).

Arsenic is found widely in the Earth's crust as sulphides, or metal arsenides or arsenates. It is usually present in natural waters at concentrations less than 1–2 mg dm⁻³ [12], but in ground water, its concentration can be significantly elevated. Many studies have shown that long-term exposure to arsenic in drinking-

water is related to increase risks of cancer in the skin, lungs, and kidneys [12]. In analysed samples arsenic was not detected.

Iron is found in natural fresh waters, especially in ground waters. Ground water containing soluble iron may remain clean when pumped out, but exposure to air may cause precipitation of iron due to oxidation, which causes brownish-red colour. Iron affects target organs such as liver, cardiovascular system and kidneys [16]. All water samples were with iron content below the allowed limits ($4.4\text{--}168.3 \mu\text{g dm}^{-3}$).

Manganese concentration levels ranged from 2.2 to $8.1 \mu\text{g dm}^{-3}$ and met all the set guidelines. At higher concentration levels (higher than 0.1 mg dm^{-3}), manganese causes an undesirable taste in drinking-water, and precipitates due to oxidation of Mn(II) to Mn(IV) resulting in stain household utensils and clothes [17]. The presence of manganese in drinking-water may also lead to the accumulation of deposits in the distribution systems [12].

Cadmium has carcinogenic properties as well as long biological half-life, leading to chronic effects as a result of accumulation in the liver [12,18]. It can also cause kidney damage, as well as produce acute health effects resulting from overexposure to high concentrations [18]. Cadmium was not detected in any of the analysed samples.

Cobalt has both beneficial and harmful effects on human health. It is beneficial for humans as a part of vitamin B12, which is essential to human health. At elevated levels cobalt provokes harmful health effects. It can cause pulmonary syndrome, skin syndrome, allergy, gastrointestinal irritations, nausea, cardiomyopathy, haematological disorders [19]. Fortunately, cobalt is rarely detected in drinking-water. According to WHO, the concentration of cobalt in drinking-water is low and ranges from 0.1 to $5 \mu\text{g dm}^{-3}$ [12]. In analysed water samples cobalt was not detected.

Chromium is present in small quantities in the environment. The toxicity of chromium depends on its physicochemical form; hexavalent salts are considered the most dangerous [20]. Prolonged consumption of water containing elevated concentrations of chromium, can damage liver, kidney and nervous tissue [21]. Chromium was not detected in any of the analysed samples.

Copper as many other metals can exist in aquatic environment in three forms, namely soluble, colloidal and particulate. At lower doses it is essential element for organisms. Excess of copper in human body is toxic and causes hypertension and produces pathological changes in brain tissues [12]. Of all analysed water samples, only sample 16 contained copper (0.015 mg dm^{-3}), but in concentration much lower than permitted.

Long-term exposure to elevated concentrations of nickel causes weight loss, heart and liver damage, and dermatitis [21]. In all analysed samples nickel was not detected.

Zinc is an essential trace mineral that is found in water and food in the form of salts or organic complexes. Although concentrations of zinc in surface and ground waters do not exceed 10 or $50 \mu\text{g dm}^{-3}$, concentration of zinc in tap water can be much higher, due to the dissolution of the element from pipes and valves materials [21]. Zinc was not detected in any of the analysed samples.

Lead occurs naturally in the environment. However, the most significant concentrations of this element found in the environment are the result of the human activities. Lead is toxic and it accumulates in kidneys and skeleton. Over centuries lead became recognised as a cumulative general metabolic poison. Lead contamination of the ground waters may result from industrial effluents, old plumbing household sewages, agricultural run-off containing phosphatic fertilizers, and human and animal excreta [16]. Lead was not detected in any of the analysed samples.

Previous similar investigations focusing on heavy metal determinations in spring and river water samples in Serbia showed the presence of cadmium, lead, zinc and nickel [22,23]. On the basis of the obtained results and by comparison with the world average contents of trace elements for unpolluted rivers, contents of heavy metals in analyzed water samples from the river South Morava were very low, classifying those river sites as unpolluted at the time of sampling. Additional experiments are in progress to provide reliable statistic results in order to examine if these alluvial deposits may be used as good sources of water supply to many communities close to this river.

With industry development the heavy metal pollution became an issue of global proportions. The fact that heavy metals did not exceed permitted limits in analysed water samples favours the quality of water from investigated area. On the other hand, some analysed water samples cannot be used for public water supply, due to calcium and magnesium level higher than permitted. These waters, however, can be used as bottled drinking waters according to Serbian regulations [7]. For such waters elevated level of specific mineral should be clearly labelled. As a result, on Serbian markets natural waters rich in different minerals (magnesium and potassium) can be found.

CONCLUSIONS

According to conducted study focusing on quality control of natural spring, river and ground waters in South-East Serbia, it can be concluded that majority of samples were free of heavy metals. The exceptions were iron, manganese, and copper found in some samples, but their contents were within allowed limits. As for major elements, including calcium, magnesium and potassium, some samples had higher contents of those specified in the guidelines. Elevated levels of calcium

were detected in two samples of spring waters and two samples of ground waters, whereas increased potassium concentration was observed in one sample of ground water. These samples, however, fulfilled the requirements of WHO in respect to specified maximum calcium and potassium levels. Levels of magnesium, higher of those specified by Serbian regulations and by WHO, were found in three ground water samples and one spring water sample. Data on the quality of unused water sources are sparse, especially for the studied region, and on the basis of obtained results a great potential for new water resources are recognized, especially for the production of bottled water. These preliminary results highlight the need for further investigations that should be focused on microbiological quality as well as organic chemical pollutants which may be present in water sources. In addition, hydrogeological assessment of abundance and assessments at periodic intervals are necessary to verify the effectiveness of management decisions of ground and spring water resources exploitation. In the meantime, to protect and preserve these water resources certain measures should be immediately taken. The preservation of water resources should be performed in economically, socially, and environmentally sustainable manner.

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IZVOD**ISPITIVANJE KVALITETA PODZEMNIH, IZVORSKIH I REČNIH VODA SA PODRUČJA JUGOISTOČNE SRBIJE**

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Ovaj rad predstavlja analizu kvaliteta prirodnih voda iz jugoistočne Srbije. Sadržaj arsena, aluminijuma, kalcijuma, kadmijuma, kobalta, hroma, bakra, gvožđa, kalijuma, magnezijuma, mangana, natrijuma, nikla, olova i cinka je određivan u izvorskim i podzemnim vodama, kao i u rečnoj vodi. Analiza navedenih elemenata je izvedena ICP-AES tehnikom. Analizirani su uzorci voda iz oblasti jugoistočne Srbije, uključujući područja planine Rtnja, Ozrena, Bukovika, Vrdenika i Čemernika, kao i područje doline reke Južne Morave. Određeni sadržaji analiziranih elemenata su poređeni sa važećim pravilnicima Republike Srbije i preporukama Svetske zdravstvene organizacije (SZO). Visoki sadržaj makro-elemenata, odnosno kalcijuma, magnezijuma i kalijuma uočeni su u izvorskim i nekoliko uzoraka podzemnih voda, što može biti posledica direktnog uticaja magmatskih stena. Pojedini uzorci voda su sadržali nešto više sadržaje gvožđa, mangana i bakra u koncentraciji do $168,3, 8,10$ i $14,9 \mu\text{g dm}^{-3}$, redom, ali u granicama propisanim Pravilnikom i u skladu da preporukama SZO. Ostali teški metali nisu detektovani u ispitivanim uzorcima. Na osnovu dobijenih rezultata se može zaključiti da testirani uzorci podzemnih i izvorskih voda imaju značajan potencijal u smislu proizvodnje flaširane vode, uz neophodna dodatna istraživanja. Dodatna istraživanja bi trebalo usmeriti na kompletну hemijsku analizu i ispitivanje fizičkih i mikrobioloških karakteristika uzorka voda. Pored toga, neophodno je sprovesti sistematsko hidrogeološko ispitivanje izdašnosti podzemnih i izvorskih voda tokom svih godišnjih doba. U međuvremenu treba preuzeti mere predostrožnosti kako bi se ovi vodeni resursi zaštitili i očuvali.

Ključne reči: Kvalitet vode • Minerali • Teški metali • Vodeni resursi • Upravljanje vodenim resursima