Multi-elemental analysis of real water samples from the Požega area, Serbia

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10.5937/zasmat2201058P

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Scientific paper
ISSN 0351-9465, E-ISSN 2466-2585
https://doi.org/10.5937/zasmat2201058P

Zastita Materijala 63 (1)
58 - 67 (2022)

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ABSTRACT

The aim of this study was to examine the quality of real samples of drinking water, collected from the territory of the city of Požega and the surrounding rural areas. An analytical technique of inductively coupled plasma with optical emission spectrometry (ICP-OES) was used to determine the concentrations of macro- and microelements in water samples. The obtained results were compared with the maximum allowed concentrations of elements specified by the national Regulation on hygienic quality of drinking water, as well as the recommendations of the World Health Organization (WHO) and the US Environmental Protection Agency (EPA). The analysed waters were low-mineralized (<1000 mg/L), calcium waters, with low sodium content. The concentrations of all tested elements in the water samples were within the allowed values. There were obvious differences in chemical composition between surface water samples and groundwater samples. All tested waters can be safely used for irrigation, because the risk of soil salinization, sodium accumulation and harmful effects of boron on crops was assessed as insignificant.

Keywords: *microelements, macroelements, water quality, ICP-OES analysis, water supply system, Požega*

1. INTRODUCTION

Water is one of the most important but also the most endangered natural resource. Over 97% of the Earth's water is ocean water, which is unusable without further processing. Regarding freshwater resources (making up less than 3% of the world's water) only 12% is available for direct use [1], either as drinking water or as household water. Apart from the continuous growth of the world's population (primarily in urban areas), which has led to increasing consumption of drinking water as a global problem, the accelerated process of urbanization and industrialization are the main reasons for endangering water resources in Serbia. Increasing amounts of water are necessary for the process of food production, as well as for obtaining electricity, which results in increased pollution of aquatic ecosystems throughout our country [2].

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Paper received: 02. 10. 2021.
Paper accepted: 05. 11. 2021.
Paper is available on the website: www.idk.org.rs/journal

Surface waters are a living space for all aquatic organisms, but they are directly jeopardized, as a consequence of the impact of municipal and industrial wastewater. In addition, a significant part of wastewaters in Serbia is often discharged into rivers and lakes without any treatment. Indirectly, the anthropogenic impact also leads to groundwater pollution through contact with the surrounding soil matrix and rocks. Most freshwaters have a similar chemical composition, which is conditioned by both natural and anthropogenic factors. In general, 5 basic groups of chemical constituents can be distinguished: basic ions (macro components, represented as cations and anions), soluble gases, biogenic substances, organic substances and microelements [3].

Knowledge of the basic physical and chemical parameters, as well as the detailed chemical composition of water, is important because it is one of the elementary ways to assess water quality. Drinking water must meet certain standards in order to be safe to use. In addition to the presence of sufficient amounts of important biogenic macro- and microelements, it is essential that water does not contain any of (potentially) toxic elements.

However, if they are present, it should be within the limits recommended by the relevant international agencies (such as World Health Organization – WHO; U.S. Environmental Protection Agency - EPA, etc.) or national legislation. Based on the hydrological data and the presence/availability of water resources, Serbia is one of the European countries with an abundance of easily accessible sources of drinking water, which currently satisfy the population needs [4]. The most recent research of the quality of water obtained investigating seven of the biggest rivers in Serbia (Danube, Sava, Drina, Tisa, Velika Morava, Zapadna Morava and Južna Morava) has demonstrated that the River Drina's water mostly complied with the national legislation requirements regarding chemical composition. It was concluded that with some minimal processing and purification, it can be used to supply the population with drinking water [5]. Such a result is valuable and atypical in the time of global industrialization and negative anthropogenic impact on water quality. Besides the use of surface water (rivers and lakes) and low-mineralized groundwater, as the main sources of water supply, mineral waters are becoming increasingly popular among the inhabitants, as a result of the increasing economic strength of the average consumer. In that sense, Serbia is characterized by a great diversity of quality mineral water springs, for example, in Vrnjačka Banja, Bukovička Banja, etc. [6]. Apart from studying mineral water sources, it is necessary to examine the quality of drinking water available in local communities, in order to determine water quality accessible for the population in different parts of our country. In that sense, data on water quality is available from the area of Zrenjanin [7], as well as from the areas of Rasina [8] and Braničevo district [9]. The importance of such "local" surveys is reflected in the fact that these studies indicated increased arsenic and boron concentrations in the water samples from the Zrenjanin area [7], as well as the microbiological pollution of certain drinking water samples from the Rasina district [8].

Accordingly, the main goal of this study was a physical and chemical analysis of drinking water samples obtained from the Požega area (Serbia) in order to determine its quality. For that purpose, a detailed elemental analysis of water samples was conducted by applying ICP-OES (Inductively Coupled Plasma-Optical Emission Spectrometry) technique. In total, 15 different elements were monitored and quantified. Obtained results were processed and compared with national and international legislative recommendations for drinking water quality. In addition, waters suitability for irrigation was assessed, based on the values of electrical conductivity, sodium absorption ratio (SAR) and boron content.

2. EXPERIMENTAL PART

2.1. Study area

The municipality of Požega belongs to the Zlatibor administrative district in Western Serbia (43°50′46″N 20°02′08″E) and covers an area of 426 km^2. In this study, real water samples from the territory of the municipality of Požega were taken from four locations (Figure 1): tap water in the village of Gornja Dobrinja (**U1**), tap water in the town of Požega (**U2**), groundwater from a dug well in Gornja Dobrinja (**U3**), and water from the drinking water basin in Gornja Dobrinja (**U4**).

Figure 1. Locations of water samples

Slika 1. Lokacije uzoraka vode

The town of Požega and the surrounding villages, including Gornja Dobrinja, are supplied with drinking water from the water supply system on the Rzav River (samples **U1** and **U2**). Additionally, several dug wells and a drinking water basin in Gornja Dobrinja provide fresh water to individual households (samples **U3** and **U4**), both drawing groundwater, which, in case of a drinking water basin, previously passes through a system of membrane filters (sample **U4**).

2.2. Analytical procedure

To determine content of the elements, water samples were taken in the polyethylene bottles of 1 L which were previously washed with HNO_3 (1:1, v/v) and then with deionized and ultra-pure water. For the purpose of element stabilization 1 mL of 65% HNO_3 (65 wt.%, Suprapur®), Merck KGaA, Darmstadt, Germany) was added to pH<2. The samples were stored in the refrigerator.

The digestion was performed on the Advanced Microwave Digestion System (ETHOS 1, Milestone, Italy) using HPR-1000/10S high pressure segmented rotor. The pressure-resistant PTFE vessels (volume 100 mL), were used in this study, consisting of the fluoropolymer liner. In the digestion, 45.00 mL sample precisely weighed was mixed in each clean vessel with 5 mL of HNO_3 (65%, Suprapure®, Merck, Darmstadt, Germany) at 160 ºC for 10 min and then heated with microwave energy for 10 min. The temperature was controlled with a predetermined power program. The temperature was typically raised to 160 ºC in the first 10 min, and to the highest temperature of 165 ºC in the next 10 min, and then cooled down rapidly. After cooling and without filtration, the solution was diluted to a fixed volume into a 50 mL volumetric flask and made up to volume with ultra-pure water (US EPA Method 3015). The content of elements in solution samples was determined by inductively coupled plasma optical emission spectrometry (ICP-OES) [7]. ICP-OES measurement was performed using Thermo Scientific iCAP 6500 Duo ICP (Thermo Fisher Scientific, Cambridge, UK).

Standards for the instrument calibration were prepared on the basis of three multi-elements and one mono-element certified reference solution ICP Standards: SS-Low Level Elements ICV Stock and ILM 05.2 ICS Stock 1 (VHG Labs, Inc- Part of LGC Standards, Manchester, NH 03103 USA) and Multi-Element Plasma Standard Solution 4, Specpure® (Alfa Aesar GmbH and Co KG, Germany). The analytical process quality control, performed by the use of EPA Method 200.7 LPC Solution certified reference material (ULTRA Scientific, USA) indicated that the resulting concentrations were within 96-104% [7].

2.3. Irrigation indices

The SAR index (sodium adsorption ratio), one of the basic criteria for assessing the suitability of water for irrigation, was calculated based on the concentrations of sodium, calcium and magnesium (in meq/L), according to the following formula [10]:

$$SAR = \frac{Na^{+}}{\sqrt{\frac{1}{2(Ca^{2+} + Mg^{2+})}}}$$

3. RESULTS AND DISCUSSION

All tested water samples were taken from the same area (Figure 1); however, differences in chemical composition are obvious (Tables 1 and 2). This is largely due to the fact that the first two samples are, essentially, surface water, partially chemically treated, to meet the criteria of drinking water, while the other two samples are groundwater. Furthermore, the **U3** groundwater sample is not subject to any quality improvement treatment (taken from a household well), and the **U4** groundwater sample undergoes filtration, before entering the drinking water basin. The most pronounced difference between the analysed waters is reflected in their mineralization, i.e. the total content of dissolved solids, which in this study was estimated on the basis of measured values of electrical conductivity (EC, in µS/cm) [11]. In all four cases, the electrical conductivity meets the criteria of the national regulations (<2500 µS/cm) [12], and these are all low-mineralized waters (mineralization <1000 mg/L) [13]. Groundwater samples (**U3** and **U4**) are distinguished by higher EC values (Table 1), as a result of more intensive water-rock contact. The described differences in mineralization can be observed by comparing the content of macro-components, i.e. the main cations of natural waters − calcium, magnesium and sodium [13]. These are elements essential for human health, necessary for the normal development of various processes in the human body: building bone tissue, activating enzymes, maintaining the tone of blood vessels, transmitting nerve impulses, etc. [14].

All analysed waters belong to the calcium group, since the content of this metal is dominant in the chemical composition (Figure 2), except that samples **U3** and **U4** contain approximately twice the concentration of calcium (139.30 mg/L and 125.90 mg/L) compared to samples **U1** and **U2** (64.22 mg/L and 67.08 mg/L) (Figure 1, Table 1). Regarding the measured concentrations of magnesium, the difference between tap water (6.83 mg/L and 5.65 mg/L) and well water (34.24 mg/L and 22.19 mg/L), is even more pronounced (Figure 2). The analysed waters are characterized by extremely low concentrations of sodium (<5 mg/L), with the exception of the well in Gornja Dobrinja (sample **U3**), where 20.28 mg/L of sodium was recorded. Interestingly enough, the World Health Organization (WHO) states that there is no solid, unambiguous evidence to support the claim that sodium in drinking water leads to hypertension, and therefore this organization does not issue recommendations regarding the maximum sodium content in drinking water [15]. The Republic of Serbia's regulation on hygienic quality of drinking water has been recently amended, so the MAC[1] for sodium is no longer 150 mg/L, but 200 mg/L [12].

Although it is not considered an essential element for humans, silicon is important for bone calcification, helps the wound healing process and it's important for optimal synthesis of collagen, a protein found in various connective tissues. The main sources of silicon are drinking water and fibre-rich foods [16]. The British Food Standardization Agency recommends an upper limit of total daily silicon intake of 1500 mg SiO_2, for an average adult [17]. There are no considerable variations in silicon concentrations among the analysed water samples (1.96–6.22 mg/L), and these concentrations are in accordance with median silicon content in European surface waters (3.75 mg/L), as well as in bottled waters found on the European market (6,5 mg/L) [18].

Potassium is an essential element for humans, because it plays an important role in numerous physiological processes: muscle contractions, normal heart function, nerve conduction, metabolism of various substances, etc. [19]. In a mixed diet, potassium is most abundant in fruits. An adult person ingests an average of 2−3 g of potassium per day through food [20,21]. The opinion of the World Health Organization (WHO) is that potassium in drinking water practically never occurs in concentrations that could pose a risk to healthy people. Therefore, the WHO does not set a recommended upper limit for the content of this metal in drinking water [15], while domestic regulations specify a MAC of 12 mg/L. The potassium content of 6.73 mg/L separates the **U3** sample from the rest of the samples, in which the concentrations of this element are several times lower (Table 1).

The dominant form of sulfur occurrence in most low-mineralized, shallow waters is the sulfate ion [22]. Although the source of sulfates in natural waters can be anthropogenic (industrial wastewater, precipitation in urban areas), the highest concentrations appear in groundwater and have a natural origin (dissolution of sulfides, sulfates and other sulfur minerals) [15]. This is reflected in the data shown in Table 1 – the maximum concentration of sulfur (37.22 mg/L) was recorded precisely in the groundwater in Gornja Dobrinja (sample **U3**).

Table 1. Basic physical and chemical parameters and concentrations of macroelements of the analyzed water samples

Tabela 1. Osnovni fizičko-hemijski parametri i koncentracije makroelemenata ispitivanih uzoraka voda

Sample	Temp	pH	EC[2]	Total hardness (TH)	NH3	Ca	K	Mg	Na	S	Si
	°C		μS/cm	mg $CaCO_3$/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L
U1	19.9	8.12	408	179	0.146	64.22	0.85	6.83	1.76	10.14	2.15
U2	18.0	7.76	354	180	0.003	67.08	0.69	5.65	1.36	10.36	1.96
U3	17.1	7.40	985	460	0.101	139.30	6.73	34.24	20.28	37.22	4.40
U4	19.2	7.32	708	370	0.049	125.90	1.74	22.19	3.23	9.60	6.22
MAC[1] (mg/L)	Source temp. or lower	6.8-8.5	2500	/	0.5	200	12	50	200	250*	/

[1]MAC – Maximum allowed concentration, in mg/L [12]

[2]The following data: temperature, pH, electrical conductivity (EC), total hardness (TH) and ammonia concentration, are an integral part of this research, and they were previously published in Maloparac and Pantelić, 2017 [23].

The presented values of basic physical and chemical parameters, as well as concentrations of macro components, meet the national regulations in all analysed water samples (Table 1)[2]. Comparing the data in Table 1 and on Figure 2, it is apparent that the groundwater from the well in Gornja Dobrinja (sample **U3**) has the highest contents of all analysed macro components, and thus the highest mineralization. Water from the drinking water basin in Gornja Dobrinja (sample **U4**) follows (also a groundwater in its origin), but its slightly lower mineralization can be attributed to partial treatment before storage in the basin, as well as to differences in depth and lithological characteristics of the aquifer.

Concentrations of all analysed microelements are within the permitted limits, defined by the national regulations [12]. However, the trend of higher contents of macro components in groundwater, compared to tap water, is not pronounced in micro components too. Concentrations of aluminium, although far below the specified MAC (Table 2), are noticeably higher in tap water (samples **U1** and **U2**) than in groundwater (samples **U3** and **U4**) (Figure 3). This might be due to the use of aluminium salts as a coagulant in the chemical treatment of drinking water [15]. The observed arsenic concentrations are far lower than the prescribed upper limit (Table 2), while the boron concentrations are below the detection limit in all tested samples. The barium content in natural waters is mostly limited by the solubility product of the mineral barite ($BaSO_4$), and due to the wide distribution of sulfate in water, elevated concentrations of barium in low-mineralized waters are rare (average for surface water is 20 μg/L) [22]. Barium concentrations in tap water in the Požega area correspond to the stated average value (Table 2), while groundwater is characterized by higher barium content (**U3** − 137.30 μg/L and **U4** − 131.20 μg/L) (Figure 3).

Cadmium is seldom found in unpolluted natural waters (average concentration in European rivers is <0.01–0.1 μg/L) [24]. Elevated concentrations of this metal are most often associated with the mine waters from abandoned mining sites, as well as waste waters of metallurgical plants [25,22]. It is an extremely toxic element, which primarily accumulates in the kidneys and is slowly eliminated from the body [26]. Significantly lower cadmium concentrations than the MAC prescribed by the national regulations were measured in all tested samples (Table 2).

Table 2. Concentrations of microelements in the analyzed water samples

Tabela 2. Koncentracije mikroelemenata u ispitivanim uzorcima voda

Sample	Al	As	B	Ba	Cd	Co	Cu	Fe	Se
	μg/L	μg/L	μg/L	μg/L	μg/L	μg/L	μg/L	μg/L	μg/L
U1	53.21	0.43	<1	16.15	0.28	0.83	5.11	1.41	5.60
U2	66.56	0.73	<1	14.54	0.25	0.69	47.03	29.84	<0.1
U3	6.05	0.75	<1	137.30	0.91	0.67	1.53	2.69	<0.1
U4	10.16	0.62	<1	131.20	0.70	<0.1	16.96	14.14	9.32
MAC (μg/L)	200	10	1000	700	3	/	2000	300	10

Cobalt is an essential trace element for humans because it is part of vitamin B_{12} [27]. Due to the lack of unequivocal evidence of a correlation between the content of this metal in drinking water and the occurrence of cancer, the US Environmental Protection Agency (EPA) did not classify cobalt as a carcinogenic element [28]. Moreover, even national regulations do not include MAC for this element [12,29]. Cobalt does not migrate well in the aquatic environment because it is easily absorbed by organic complexes and hydroxides of iron and manganese [25]. This results in a low average concentration of this element, e.g. only 0.023 μg/L in tap and bottled waters in the EU [30]. In this study, the analysed concentrations of cobalt were in range <0.1–0.83 μg/L (Table 2).

Copper is an essential nutrient for humans, but also a potential contaminant of drinking water. It is an important component of many enzymes (catalytic role) and proteins and is necessary for the normal course of iron metabolism and the formation of red blood cells [25]. The average concentration of copper in bottled water on the European market is 0.251 μg/L, while for water supply systems, also in Europe, it is significantly higher − 5.65 μg/L [30]. The reason for this is corrosion of water pipes due to which copper concentrations often increase in the process of water distribution [15]. The described phenomenon most probably contributes to a slightly increased content of copper in the Požega's water supply system (sample **U2** − 47.03 μg/L) (Figure 3), given that it's several decades old. However, in this study, all measured concentrations of copper were far below the prescribed MAC of 2000 μg/L [12].

The migration and forms of occurrence of iron in the aquatic environment are very complex and are influenced by a number of factors, primarily pH and Eh values. The average iron content in surface waters is 40 μg/L [22], and in European water supply systems − 3.21 μg/L [30]. Similar to copper, iron can also occur in drinking water as a consequence of the corrosion processes, in this case, involving steel water pipes [15]. This was observed in the water sample **U2**, from the Požega water supply system, with iron concentration 29.84 μg/L. All analysed water samples contained iron concentrations within the limits prescribed by the regulations of the Republic of Serbia (Table 2). Iron is an essential element for humans, with several vital functions in the body, as it is an important constituent of hemoglobin and numerous enzyme systems in various tissues [25].

Selenium is an essential microelement for humans − it protects the body from oxidative stress, participates in the regulation of the thyroid gland, and contributes to the proper metabolism of vitamins *C* and *E*, as well as coenzyme *Q* [25]. Cereals, meat, and fish are the primary sources of selenium intake, while the contribution of drinking water is generally small. Deficiency of this element often occurs among the elderly population, but also in certain parts of the world, including Western Europe [15]. If contaminants from various industrial plants are excluded, the primary origin of selenium in groundwater is from sediments, and elevated concentrations can also occur as a consequence of the use of certain types of fertilizers [31]. This could be the reason for a slightly elevated concentration of selenium in sample **U4** (9.32 μg/L, close to MAC for Se of 10 μg/L) (Table 2). On the other hand, the content of selenium in samples **U2** and **U3** was below the limit of detection, which leads to the conclusion that sediment erosion is not the source of this element in the study area.

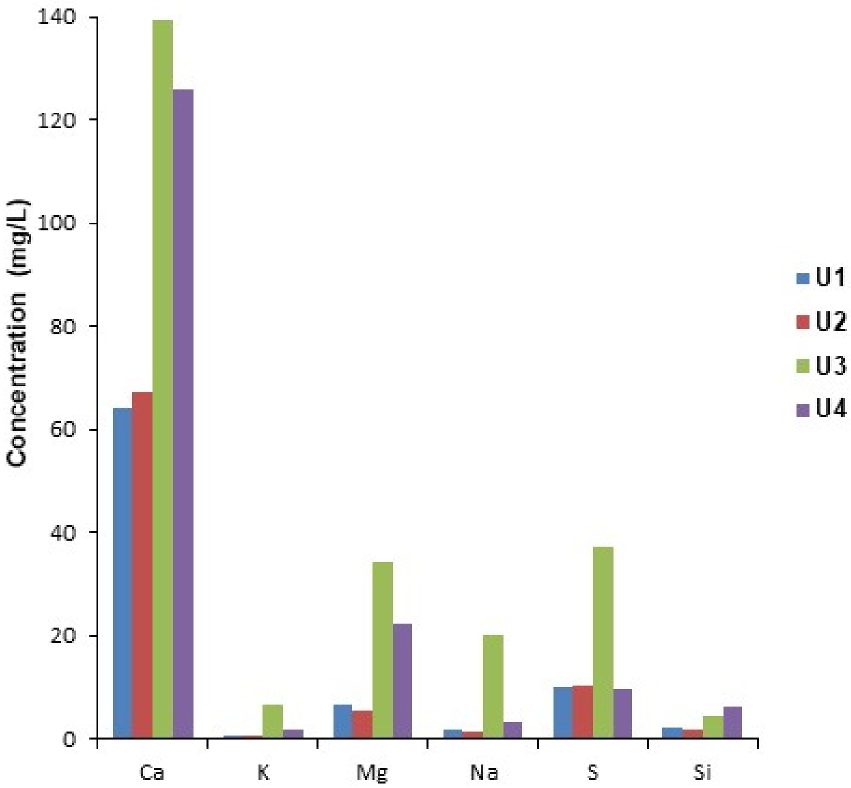

Figure 2. Comparative display of macroelements concentrations (in mg/L) in analyzed water samples (labeled U1–U4)

Slika 2. Uporedni prikaz koncentracija makroelemenata (izraženih u mg/L) u ispitivanim uzorcima voda (obeleženi sa U1–U4)

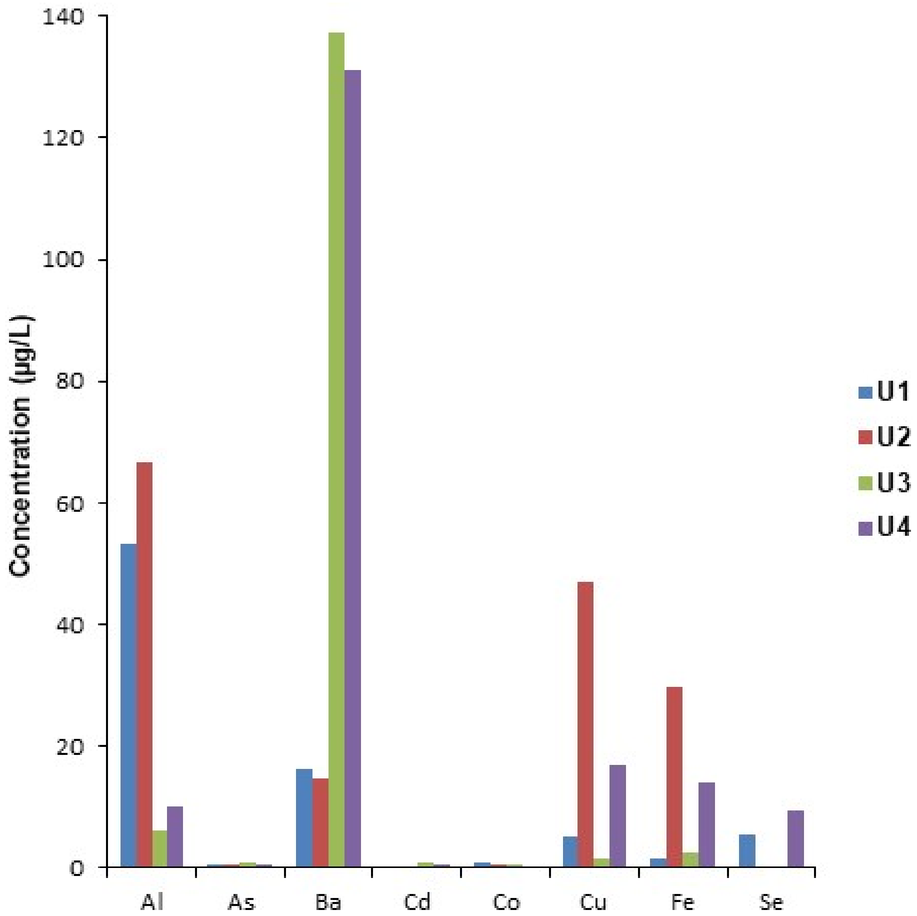

Figure 3. Comparative display of microelements concentrations (in µg/L) in analyzed water samples (labeled U1–U4)

Slika 3. Uporedni prikaz koncentracija mikroelemenata (izraženih u µg/L) u ispitivanim uzorcima voda (obeleženi sa U1–U4)

Table 3. Evaluation of the tested waters suitability for irrigation

Tabela 3. Ocena pogodnosti ispitivanih voda za navodnjavanje

Sample	Location	EC (µS/cm)	SAR	B (µg/L)
U1	water supply, G. Dobrinja	408	0.056	<1
U2	water supply, Požega	354	0.043	<1
U3	well, G. Dobrinja	985	0.399	<1
U4	basin, G. Dobrinja	708	0.070	<1
Limit values and possible effects on plants		<750: no hazard, 750-1500: some hazard, 1500-3000: moderate hazard, 3000-7500: severe hazard [34]	0-10: low sodium hazard, 10-18: medium sodium hazard, 18-26: high sodium hazard, >26: very high sodium hazard [33]	<500: satisfying for all crops, 500-1000: satisfying for most crops, 1000-2000: satisfying for semi-tolerant crops, 2000-4000: satisfying for tolerant crops only [34]

Considering that there are rural households and agricultural production in the study area, suitability of water for irrigation was also assessed. The evaluation was performed on the basis of the following parameters: 1) electrical conductivity, indicating the risk of soil salinization; 2) SAR index, indicating the sodium hazard to the soil; and 3) boron concentrations, indicating the possibility of harmful effects on plants [32,33]. Based on the values of all three applied criteria (Table 3), the use of analysed waters for irrigation purposes does not pose a risk to cultivated crops or arable land. The exception is groundwater from well in Gornja Dobrinja (Table 3), which increased electrical conductivity could be an inconvenience to certain types of crops.

4. CONCLUSION

Access to quality drinking water is one of the imperatives of healthy living. Providing such a water to a wider circle of consumers is the basic goal of any public water supply system. If the physical, chemical and microbiological safety of drinking water is routinely monitored, and the water supply network is regularly controlled and maintained, a system is safe. A detailed survey of the macro and micro elemental composition of real samples of drinking water, collected on the territory of the city of Požega and in the surrounding rural settlements, was conducted in order to assess the usability of water. The results of the ICP-OES analysis, tabulated and graphically presented in this paper, are interpreted in accordance with national regulations, as well as with the recommendations of the WHO, EPA and other international regulatory institutions.

All analyzed samples indicated low-mineralized (<1000 mg/L), calcium type waters, with low sodium content. Groundwater samples from the Gornja Dobrinja (**U3** and **U4**) are characterized by higher values of electrical conductivity and higher calcium and magnesium content, compared to tap water samples (**U1** and **U2**), which are derived from surface water. Sample **U3**, from a dug well, particularly stood out, with its highest contents of sodium, potassium and sulfur, as well as the highest electrical conductivity. In contrast, tap water is characterised by higher aluminium content, compared to groundwater, most likely due to the use of this metal as a coagulant in the chemical treatment of water. Emphasized content of iron and copper in the water supply of the city of Požega, occurs as a consequence of old, steel and copper plumbing, in contrast to significantly lower concentrations of these elements in the recently built Gornja Dobrinja's water supply system, with a distribution network made of PVC materials. The concentration of selenium in the drinking water basin in Gornja Dobrinja is at the upper limit of national legislation recommendation, possibly as a consequence of the local use of fertilizers containing selenium.

All examined water samples, regardless of their origin, were in line with national drinking-water legislation, in terms of content of macro- and microelements. In addition, the analysed waters can be safely used for irrigation, since the risk of soil salinization, sodium accumulation and the harmful effects of boron on crops, was assessed as insignificant.

Acknowledgements

This work was done within the agreement between the Faculty of Agriculture University of Belgrade and the Ministry of Education, Science, and Technological Development of the Republic of Serbia (Grant No. 451-03-9/2021-14/200116)

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IZVOD

MULTI-ELEMENTALNA ANALIZA REALNIH UZORAKA VODE SA TERITORIJE POŽEGE, SRBIJA

Cilj ovog rada bio je ispitivanje kvaliteta realnih uzoraka vode za piće, prikupljenih sa teritorije grada Požege i okolnih seoskih naselja. Za određivanje koncentracije makro- i mikroelemenata u uzorcima vode korišćena je analitička tehnika induktivno kuplovana plazma sa optičkom emisionom spektrometrijom, ICP-OES. Dobijeni rezultati upoređivani su sa maksimalno dozvoljenim kocentracijama elemenata propisanim nacionalnim Pravilnikom o higijenskoj ispravnosti vode za piće, kao i preporukama Svetske zdravstvene organizacije (WHO) i Američke agencije za zaštitu životne sredine (EPA). Analizirane vode su malomineralizovane (<1000 mg/L), kalcijumskog tipa i sa niskim sadržajem natrijuma. Sadržaj svih ispitivanih elemenata u uzorcima vode bio je u granicama dozvoljenih vrednosti. Konstatovano je da postoje očigledne razlike u makro- i mikrokomponentnom hemijskom sastavu između uzoraka površinske vode i uzoraka podzemne vode. Ispitivane vode mogu bezbedno da se koriste u meliorativne svrhe, jer je rizik od zasoljavanja zemljišta, nagomilavanja natrijuma i štetnog uticaja bora na biljne kulture procenjen kao neznatan.

Ključne reči: *mikroelementi, makroelementi, kvalitet vode, ICP-OES analiza, vodovodni sistem, Požega*

Naučni rad

Rad je primljen: 02. 10. 2021.

Rad je prihvaćen: 05. 11. 2021.

Rad je dostupan na sajtu: www.idk.org.rs/casopis