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Research article

Investigation of the Content of Heavy Metals in Water Sources of Kharkiv City, Ukraine

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Abstract

Keywords

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water objects;
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The negative impact of the anthropogenic factor on water bodies, including those ones located within urban ecosystems is analyzed in this paper. The legislative support of the specific requirements for water quality in different countries is considered. The purpose of the work is to investigate the qualitative and quantitative state of water from individual sources located in an urban ecosystem and determine its suitability for the consumption. The Shatylovsky spring, the Karpovsky spring, the spring near the Nemyshlya river, located within the Kharkiv city (Ukraine), were researched. Heavy metal identification was carried out using X-ray fluorescence analysis. It was found that all samples contained basically the same set of basic elements - strontium, copper, iron and chromium. Individual differences were associated with the presence of different amounts of tungsten, zinc, gallium, selenium, bromine, etc. in all samples. It was found that in the samples, the lead content reached 0.015 mg/dm^3 (Karpovsky spring), nickel 0.007 mg/dm^3 (Shatylovsky spring), and manganese 0.205 mg/dm^3 (the spring near the Nemyshlya river). The copper content ranged from 0.029 to 0.154 mg/dm^3, iron from 0.041 to 0.456 mg/dm^3, and chromium from 0.015 to 0.065 mg/dm^3. The obtained results make it possible to more efficiently manage water resources within the urban systems studied and indicate the need for additional water purification of all natural sources studied before using the water from those sources for drinking purposes.

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1. Introduction

The human impact on the environment has recently reached alarming levels despite the influence of natural factors, which also have a negative physical, chemical and microbiological impacts on the environment [1, 2]. Such negative changes concern both water resources and the state of air and soil [3-5] and biotic components [6]. Water objects are one of the most vulnerable components of the environment [7, 8]. In addition to this, they are significantly contaminated by micro plastics [9-11], heavy metals [12, 13], pharmaceutical substances [14, 15] and other pollutants. In turn, polluted water can be a source of pollution for other components of the environment [16].

Water bodies, especially those ones located within urban ecosystems [17, 18], need considerable attention [19]. Due to the fact that they are influenced by many negative factors [20, 21], their use as drinking and recreational resources can be dangerous or have certain limitations. In urban ecosystems, there is the influence of motor transport [22], various industries [23, 24] and surface runoff [25]. The situation is worsening because of pollutants entering the environment [26]. Emergencies such as fires and fire extinguishers are additional factors in environmental pollution, including surface runoff and water bodies in cities [27, 28].

Given the growing demand of the population for quality of drinking water, the issue of water quality is reflected in a number of national and foreign regulations. For example, WHO has developed a set of universal guidelines on water quality and human health as an approach to prevent water consumers from risks, and to set criteria for independent surveillance of the implementation of water safety regulations. Furthermore, their guidelines can be used as a basis for national legislation [29].

The EU has an extensive system of regulations that establishes uniform rules for the provision of drinking water that are in accordance with generally accepted principles and standards. Thus, the European Water Charter establishes the principles of European policy on managing water resources in order to preserve the quantity and quality of water suitable for humans, animals and plants [30]. The purpose of Council Directive 98/83/EU “On the quality of water intended for human consumption" is to protect human health from the harmful consequence of any contamination in the water intended for human consumption by ensuring its safety and purity [31].

In turn, the Constitution of Ukraine proclaims the obligation of the state to guarantee everyone environmental safety and gives the right to live healthily. In order to implement these constitutional provisions, the legislation of Ukraine (Water Code of Ukraine, Subsoil Code of Ukraine, Laws of Ukraine "On Environmental Protection", "On Ensuring Population Sanitary Conditions Against Epidemic", "On Drinking Water, Drinking Water Supply and Sewerage", "On Strategic Environmental Assessment") is designed to create mechanisms to ensure the proper quality of water intended for consumption. As a result, to control the quality of water in Ukraine a specific standard DSaNPiN 2.2.4 - 171.10, "hygienic requirements for drinking water intended for human consumption", is applied [32].

An additional factor for the unification of drinking water quality requirements was the signing of the Association Agreement between Ukraine, on the one hand, and the European Union, the European Atomic Energy Community and their Member States, on the other hand, which requires Ukrainian national regulations to be fulfilled in accordance with international and European standards in supplying safe drinking water.

However, systemic problems such as lack of financial resources, which are necessary for the proper service and maintenances of the water supply and sewerage systems, unsatisfactory technical condition of the facilities and the equipment, and the imperfect management of this industry, and so on have slowed down the adoption and implementation directives such as Council Directive 98/83/EU "On water quality intended for human consumption", Council Directive 91/271/EEU "On urban waste water purification", AND Directive 91/676/EU "On water protection against pollution caused by nitrates from agricultural sources", etc. However, measures to implement Directive 91/676/EU have not yet been implemented. The analysis of the current state of nitrate pollution from agricultural sources is underway, and a common approach to identify the areas which are vulnerable to nitrate accumulation is being agreed upon. Furthermore, the introduction of legal regulations for identifying "vulnerable zones" areas, which are areas from which nitrates flow into the water bodies and accumulate there, helps to reduce and prevent nitrate pollution [33].

A clear example of implementing the international and European standards as per drinking water supply come from Scandinavian countries. Denmark, Norway, Finland and Sweden introduced uniform rules to ensure the safety for human health of materials used in distributing and supplying water to consumers, including the production of food products. For example, in Norway, there have been the establishment of the Law on Food Production and Food Safety, the Law on Health Care and Social Readiness, the Law on Health Care, the Law on Municipal Health Services, the Decree Of the Government "On the water supply intended for human consumption". The latter regulation, which regulates the "Drinking Water Rules", sets requirements for the safe supply of sufficient amounts of useful, transparent, odorless, tasteless and colorless water intended for human consumption. In particular, the "Drinking Water Rules" stipulate the following: materials in the transport system, household distribution system and treatment facilities, etc., which are in direct or indirect contact with water, must not emit into the water substances that may harm human health or lead to a change in the specific composition of water, including the deterioration of the sensory characteristics of water [34].

In Sweden, a number of normative decrees, and in particular, the Law on General Water Supply Services, the Law on Planning and Construction, the Law on Accreditation and Technical Control, the Environmental Code, and the Regulations on Drinking Water, set limits on the concentration of harmful substances for human health that may enter the drinking water from materials used to transport the water to consumers [35]. Similar regulations as per water quality monitoring are enshrined in Finnish legislation (Law on Water Services, Law on Water, Law on Land Use and Development, Law on Approval of Certain Construction Materials, National Building Code, etc.) and Denmark (Law "On Construction", Executive Order "On Building Regulations") [36].

The information mentioned above emphasizes the relevance of the question being researched. In Ukraine, the state of natural water sources located in urban ecosystems has not been sufficiently studied. The increasing anthropogenic external force (traffic, urbanized surface runoff, industry) affects the state of water bodies in cities [25, 37]. In large cities, particularly, in Kharkiv, there is a lack of underground and surface water sources to provide residents with high-quality drinking water [38]. As a result, the state of available water sources requires more careful study to effectively manage water resources in the city. The purpose of this work is to investigate the qualitative and quantitative state of water found in sources located in the urban ecosystem and determine its suitability for consumption. The research was carried out on a number of water sources in one of the largest cities in Ukraine - Kharkiv.

Kharkiv region is one of the scientific, industrial and agricultural regions in Ukraine. As a result, its water bodies are under significant anthropogenic pressure [39]. Kharkiv is the center of Kharkiv region which is a powerful scientific and industrial cluster and its population is more than one million inhabitants. Thus, there is significant car traffic. This affects the quality of drinking water from natural sources in Kharkiv. The presence of pollutants is a negative factor affecting the quality of life of the population. As a result, more detailed information on the quality of water in the sources of the city of Kharkiv is needed.

In large megalopolises with a developed industry, of which the city of Kharkiv is an example, there is a technogenic ingress of significant amounts of heavy metals into the environment [40]. These pollutants pose an increased risk factor for human health. They cause diseases such as cirrhosis of the liver, anemia, diseases of the gastrointestinal tract, etc. [41]. Furthermore, heavy metals, may present in combination, and have cumulative effects [42]. Therefore, in this work, we focused primarily on the determination of heavy metals in drinking water.

There are various methods for determining heavy metals such as atomic absorption spectroscopy (AAS), inductively coupled plasma atomic emission spectroscopy (ICP-AES), conductometry, bioindication and so on. The method of atomic absorption spectroscopy is often used. For example, mercury (Hg), arsenic (As), lead (Pb), zinc (Zn) and cadmium (Cd) were identified in drinking water of two small-scale mining communities in Northern Ghana using AAS [43]. There was an excess of a number of these elements according to the WHO standards in each of the studied mining communities. According to the authors, anthropogenic influence was the cause of the excess.

An AAS (200A flame Atomic Absorption Spectrometer) was used to analyze water in the Ureje Water Reservoir [44]. The results indicated an excess of iron according to WHO standards, and the presence of trace amounts of other heavy metals. Anthropogenic input from the city of Ado Ekiti was noted as a factor of influence. In another work [45], researchers used the AAS method (AAS Buck 200, Germany) to determine trace amounts of heavy metals in groundwater sources from three different residential areas within Ibadan metropolis, southwest Nigeria. The results showed that high concentrations (against WHO standards) of Zn, Fe, Pb and Cd in 60%, 86.7%, 100% and 100% of groundwater samples, respectively, and the conclusions contained recommendations for purify the water before use. Geogenic and anthropogenic sources were indicated as reasons for the excess. However, despite the high sensitivity of AAS, the AAS method does not allow determination of the entire spectrum of elements at once, and the number of determined elements depends on the number of spectral lamps.

One of the most common methods for analyzing drinking water is X-ray fluorescence spectrometry. For example, researchers [46] investigated the elemental composition of drinking water with preliminary solid-phase concentration of the sample on a special disk. Another work [47] was about the potential of portable X-ray fluorescence (PXRF) to quickly screen water samples for heavy metals and/or other pollutants. The XRF method makes it possible to simultaneously determine the qualitative composition of a sample and to quantitatively determine a wide range of elements in it. An additional attractive factor is its expressiveness, simplicity and speed of sample preparation. Considering all the above, studies of water samples were carried out to determine heavy metals in this work.

2. Materials and Methods

The researched water bodies were the water bodies of Kharkiv as Karpovsky spring (near 90 Kontorskaya Street), a spring near the Nemyshlya river (near 60 Lva Landau Street), and Shatylovsky spring (near 20 Sarzinskaya Street) (Figure 1). The tap water of the city of Kharkiv (12 Chkalova Street) was used as a reference sample. Samples were taken twice within two years in the springtime.

The samples were taken in spring, because the spring surface runoff in the urban ecosystem of Kharkiv, like in any large city, is characterized by the removal of pollutants accumulated during the winter period. Accordingly, the concentration of pollutants during this period will be maximum which is increasingly dangerous to use the investigated natural sources of water for drinking purposes.

Gravimetric and X-ray fluorescence methods of analysis were used as research methods. X-ray fluorescence analysis (XRF analysis) allows the determination of the elemental composition of the sample and the gross metal content in it. This method is based on the stimulation of the characteristic radiation of the elements contained in the test sample.

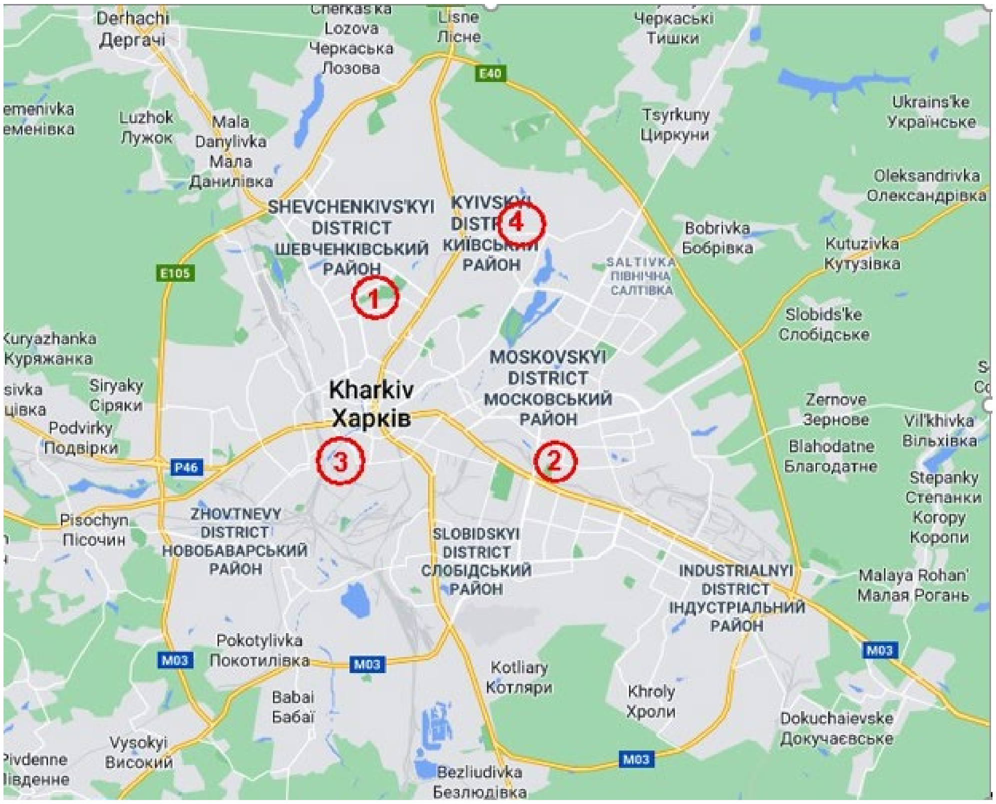


Figure 1. Water sampling locations: 1 - Shatylovsky source, 2 - Source near the Nemyshlya river, 3 - Karpovsky source, and 4 - tap water
Note: Applied from Google map

The heavy metal and trace element content of the water samples was determined using the X-ray fluorescence method (with X-ray fluorescence analyzer "Spectroscan"(Russia)), for a wavelength range of 850 milliangstroms (mÅ) - 2230 mÅ, in scanning mode with a step 4 mÅ). Analysis of these elements was performed in the dry residue of the water samples being tested. The number of samples for analyzing each sample was n = 3. Samples were taken according to water sampling guidelines [32, 48].

Disposable plastic dishes were used for sampling. The dishes used in the analysis were thoroughly washed and dried. Each sample was evaporated in a DZKW water bath (Chine), and a dry residue was obtained, which was weighed on a VLR-200 balance (Russia). After weighing, each sample of the dry sediment of the water samples were examined on a "Spectroscan" X-ray fluorescence analyzer.

The statistical process of the obtained data was carried out according to well-known statistical approaches [49] for probability P = 0.95. The mean values of the concentration were calculated and the relative standard deviation of the arithmetic mean Sr was calculated.

The reliability of the data obtained was ensured by the observance of standard sampling and sample preparation procedures [32, 48], the correct use of measuring instruments and equipment, a high level of specialists, and standard statistical processing of the obtained results [49].

3. Results and Discussion

This paper examined the water sources of Kharkiv that are used by the population for drinking, in particular, the Karpovsky spring, spring near the Nemyshlya river, the Shatylovsky spring (Figure 1). All of them belong to the Uda river [50] and they are located in the residential areas of Kharkiv.

Initially, the value of the dry residue for the studied water samples (which is often identified with the total salt content) was obtained. The obtained values are shown in Table 1. Taking into account, the dry residue *C* (mg/dm^3) is related to the electrical conductivity æ (μS) ratio:

$$æ \cdot A = C \tag{1}$$

where *A* – is a numerical factor that depends on the type of water sample and is equal to the average 0.65 [51], we can use the EU standards for the drinking water [31] to obtain the normative indicative value of the dry residue.

The normative values being compared, we can see that all samples met the requirements of national and European standards, although in the cases of the Shatylovsky spring and the Karpovsky source water samples, there are values in excess of WHO standards [29]. This may be caused by the peculiarities in the geological structure of the territory of the city of Kharkiv [38].

Then, an X-ray fluorescence analysis of the obtained dry residues of the water samples was performed. The determination of heavy metals was carried out with qualitative and quantitative parameters. At the first (qualitative) stage, the analytical spectra of samples characterizing the presence of certain elements in the sample were obtained. Figures 2-5 show general views of the spectra, on which the main elements were indicated and detected. The scale of the images is selected in a way to fully display all the main elements presented in the spectrum. It should be noted that relatively high peaks of copper are of hardware origin associated with the design features of the X-ray tube. When quantitative measurements were carried out, this factor was taken into account. It should also be taken into account that the spectra do not display two main metals - calcium and magnesium - since their analytical wavelengths are outside the measurement range of this technique.

According to Figures 2-5, all the samples have the same set of the elements - strontium, copper, iron. Also, X-ray fluorescence analysis showed the presence of chromium in all samples (the peak is not visible in the scale shown in Figures 2-5). Simultaneously, there are significant differences. First of all, it should be noted that all the samples contain different amount of strontium. Strontium (Sr) is a natural chemical analogue of calcium. Therefore, its variable presence is natural where calcium and magnesium carbonates are present as salts of water hardness. The water sample from the Shatylovsky spring (Figure 2) contained the following heavy metals such as copper (Cu), tungsten (W) and a small amount of iron (Fe). Of the lighter elements, strontium (Sr), bromine (Br), selenium (Se) predominated in the spectrum of the dry residue of the sample.

The water sample from the Karpovsky spring (Figure 3a), like the previous sample, contained the heavy metals such as copper (Cu), zinc (Zn), tungsten (W), as well as a small amount of iron (Fe). A rather significant peak for bromine (Br) should be noted. Traces of some elements are visible in the detailed spectrum (Figure 3b), in particular, the sample contains selenium (Se) and gallium (Ga). The water sample taken from the source near the Nemyshlya river (Figure 4) contained the elements such as strontium (Sr), bromine (Br), copper (Cu), and a small amount (Fe). The reference sample of tap water (Figure 5) contained elements such as strontium (Sr), bromine (Br), copper (Cu), iron (Fe), as well as traces of zinc (Zn).

Thus, the qualitative analysis carried out in some samples showed microquantities of such elements as tungsten, zinc, gallium (W, Zn, Ga), etc. However, in the investigated samples, their content was below the determination range of the quantitative analysis method. Therefore, henceforth, these elements were not determined.

Table 1. Results of determination of dry residue in water samples

No.	Sample name	Dry residue, mg/dm^3				
		Season 1	Season 2	Ukrainian standard [32]	EU standard [31]	WHO standard [29]
1	Shatylovsky source	1006.4	715.8			
2	Source near the Nemyshlya river	430.8	596.4	1500	1625	1000
4	Karpovsky source	1007.8	1194.8			
3	Tap water	561.6	696.2			

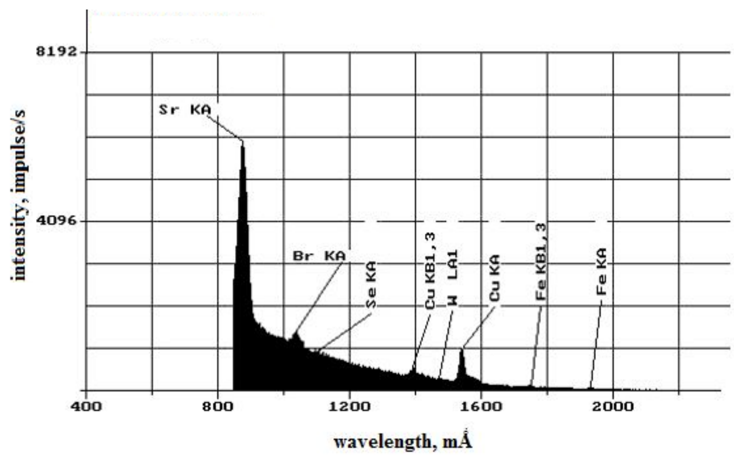
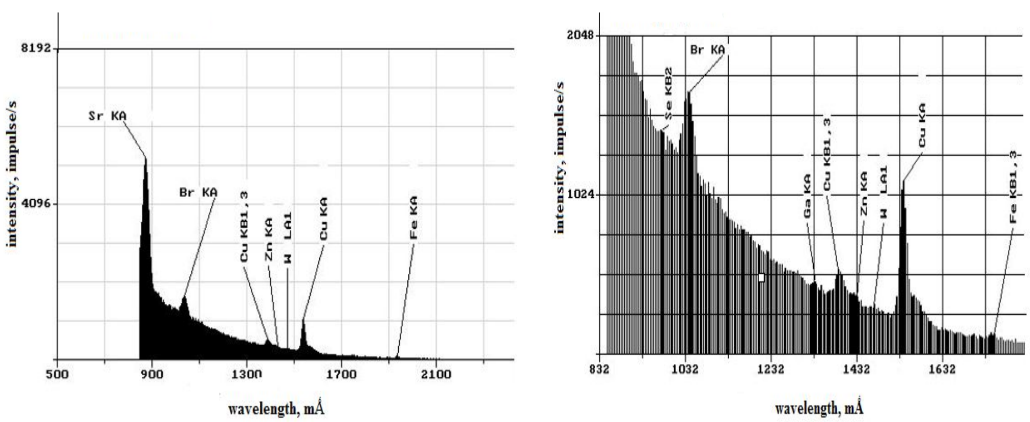


Figure 2. X-ray spectrum of the water sample from the Shatylovsky source



(a) **(b)**

Figure 3. X-ray spectrum of the water sample from the Karpovsky source (a) and the magnified part of the X-ray spectrum (b)

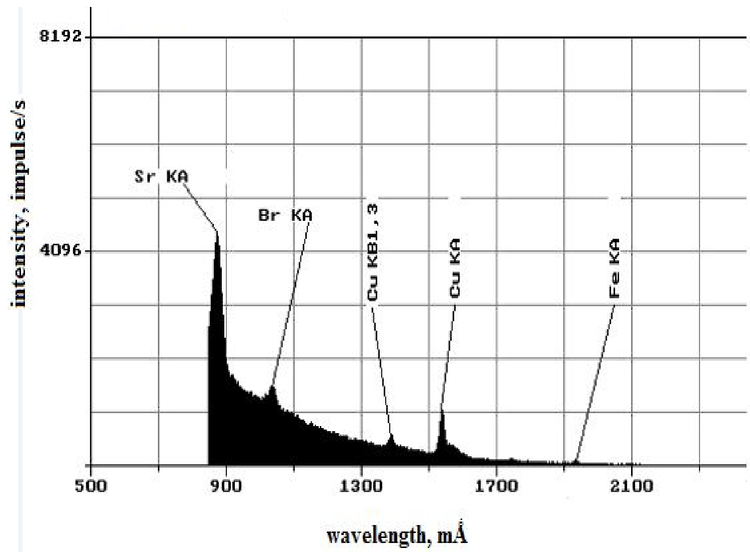


Figure 4. X-ray spectrum of the water sample from the source near the Nemyshlya river

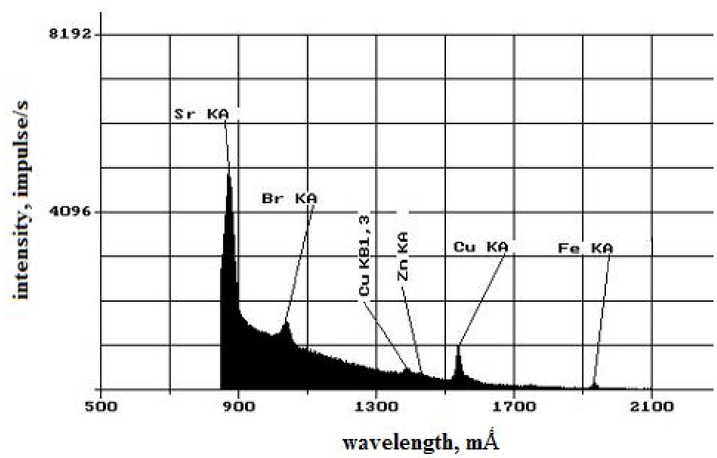


Figure 5. X-ray spectrum of the tap water sample

In the second (quantitative) stage of the study, the water samples were analyzed to determine such heavy metals as Pb, Cu, Ni, Fe, Mn, Cr. The results of the quantitative analyses of the water samples are given in Table 2. According to the obtained data, there is a discrepancy between water from the Karpovsky spring and the spring near the Nemyshlya river as per the requirements of the national and foreign standards.

Table 2 shows that the content of lead, which is one of the most toxic metals, did not go beyond the standards of national and foreign documents, and its content was noted in the water samples taken from the Shatylovsky and Karpovsky springs, as well as in the tap water. However, it should be noted that the values of these lead concentrations are on the verge of the sensitivity of

Table 2. The content of elements in the studied water samples (mg/dm³)

Element	Season	Shatylovsky source	Karpovsky source	Source near the Nemyshlya river	Tap water	National standard [32]	EU standard [31]	WHO standard [29]
Pb	Season 1	0.014	0.015	n/d	n/d	0.01	0.01	0.01
	S_r, %	58.2	58.2	-	-			
	Season 2	n/d	0.005	n/d	0.003			
	S_r, %	-	58.2	-	58.2			
Cu	Season 1	0.050	0.102	0.042	0.029	1.0	2.0	2.0
	S_r, %	58.2	35.9	58.2	8.6			
	Season 2	0.033	0.154	0.135	0.050			
	S_r, %	58.2	2.0	3.1	58.2			
Ni	Season 1	0.007	0.003	n/d	0.004	0.02	0.02	0.07
	S_r, %	17.3	13.3	-	31.5			
	Season 2	0.001	n/d	n/d	0.002			
	S_r, %	58.2	-	-	58.2			
Fe	Season 1	0.082	0.121	0.456	0.053	0.2	0.2	-
	S_r, %	10.7	47.8	12.1	20.5			
	Season 2	0.147	0.041	0.075	0.473			
	S_r, %	8.7	31.3	23.3	8.3			
Mn	Season 1	n/d	0.012	0.012	0.025	0.05	0.05	-
	S_r, %	-	58.2	58.2	7.3			
	Season 2	0.004	0.017	0.205	0.031			
	S_r, %	58.2	24.3	7.1	4.0			
Cr	Season 1	0.017	0.065	0.035	0.025	0.05	0.05	0.05
	S_r, %	58.2	15.7	15.4	58.2			
	Season 2	0.015	0.0147	0.038	0.039			
	S_r, %	47.8	15.6	20.6	20.8			

the applied technique. Therefore, the obtained data should be considered as approximate. High errors in the determination of a number of elements in the water samples were due to the fact that their contents were at the detection limit of the method. It should also be noted that trace elements of Cu, Ni, Cr were observed in the separate water samples in the 1st and 2nd seasons.

At the same time, it was seen that the purest water sample from the Shatylovsky spring was comparable to the tap water by the content of several metals or, better (the quality in relation to iron), whereas the Karpovsky spring and the source near the Nemyshlya river have some excess in the content of chromium (Karpovsky spring), iron (the source near the Nemyshlya river) and manganese (the source near the Nemyshlya river) in the 1st or the 2nd studied seasons. Probably, such excesses were due to the close location of the industrial zone (the source near the Nemyshlya river) and a railway (the Karpovsky source), from which the surface runoff caused these metals to enter these sources.

The probable reason for the rather high iron content in the tap water (Table 2) is worn out steel pipes and their corrosion [52] in the city's water supply system. The content of lead, copper and nickel in the studied sources was below the national standard values, the European standards, and WHO standards [29, 31, 32], or these metals were not detected.

While iron, manganese and chromium were present in almost all the water samples during this period, and in some cases, the excess in accordance with the national and foreign water quality standards.

The climate of the area, in which the city of Kharkiv is located, is characterized by the seasonality of precipitation [53-55]. Obviously, the increase in the amount of precipitation in the spring contributes to a greater washout of pollutants, including heavy metals, with surface runoff and their transfer to groundwater, and then to natural springs in the city of Kharkiv.

It should be noted that there is a seasonal tendency towards the increase in copper content, with the exception of the Shatylovsky spring. For iron, a similar situation was observed only for the Shatylovsky source. This feature of the Shatylovsky spring was probably due to the deeper occurrence (up to 300 m) of the aquifers, and, consequently, the less influence of the surface runoff, whereas the aquifers of the source near the river Nemyshlya (up to 30 m) and Karpovsky spring (up to 60 m) were more susceptible to the influence of surface runoff from the city of Kharkiv.

4. Conclusions

The quantitative and qualitative analysis of the water from some sources located within the urban ecosystem, the city of Kharkiv, was carried out. It was noted that in accordance with dry residue parameter, all the water samples met the requirements of national and European standards, although in the Shatylovsky source and the Karpovsky sourcesample was higher than the WHO standards.

The qualitative elemental XRF analysis showed the presence of various elements in all the water samples under the study, i.e. strontium, copper, iron and chromium. Individual differences were associated with the presence of different amounts of strontium in all the samples, as well as for the trace elements: tungsten, zinc, gallium, selenium, and bromine.

The quantitative XRF analysis of the water samples as per the content of Pb, Cu, Ni, Fe, Mn, Cr showed the presence of small amounts of lead in all the samples, except for the spring water near the Nemyshlya river. In some samples the lead content reached 0.015 mg/dm^3, nickel was 0.007 mg/dm^3, manganese was 0.205 mg/dm^3, and the copper content ranged from 0.029 to 0.154 mg/dm^3, iron is from 0.041 to 0.456 mg/dm^3, and chromium from 0.015 to 0.065 mg/dm^3.

The water from the Karpovsky spring and the source near the Nemyshlya river had some values in excess of the national and foreign standards for the content of chromium (the Karpovsky spring), iron (the source near the Nemyshlya river) and manganese (the source near the Nemyshlya river) in the 1^{st} or 2^{nd} studied seasons, which was probably due to the proximity of the industrial zone (the source near the Nemyshlya river) and the railway (the Karpovsky source).

In one of the samples of the tap water in Kharkiv, the iron content was increased, probably due to the deterioration of equipment used in the water supply system. The water of the Shatylovsky spring fully complied with the national and foreign water quality standards as per the content of the metals studied. In the future, it is recommended that officials carry out seasonal monitoring of the water quality of the natural sources in the city of Kharkiv. When people use water from all the investigated natural sources for drinking purposes, preliminary purification is necessary. The obtained results will be useful for the end users of the city of Kharkiv in order to choose and use their water source wisely, and to assist local authorities to rationally manage the water resources.

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