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MACROBIOGENIC ELEMENTS IN PACKAGED AND GROUNDWATER WATERS FROM VARIOUS SLOVAK AND FOREIGN REGIONS

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ABSTRACT

In the work, the contents of macrobiogenic elements (Na, K, Ca, Mg) in 20 randomly selected samples of bottled water sold in commercial networks in the Slovak Republic were investigated, and these values were compared with our analyses of the same macroelements in 8 groundwater samples. The aim of the work was, except of determining the contents of the elements in the waters, also finding the existence of a mutual correlation between their contents and indirectly analysing of the geochemical composition of the rocks that are in contact with these waters. Bottled water samples obtained from retail chains originated from the Slovak Republic and also from abroad: from the Czech Republic, Austria, France and Hungary - all these monitored regions belong to the Alpine-Himalayan system from a geomorphological point of view. The highest contents of calcium and magnesium were assessed in bottled waters *Gemerka* (Banská Bystrica region), *Salvator* and *Baldovská* (Prešov region). High content values of sodium were determined in bottled waters *Budiš* and *Fatra* (Žilina region) and high contents of potassium in bottled waters *Budiš* (Žilina region) and *Salvator* (Prešov region). In contrary, the lowest contents of calcium and magnesium were determined in bottled waters *Oravská* (Žilina region) and in bottled water *Aro* from Czech republic. In bottled waters *Zlatá studňa Jasenovo*, *Rajec* (Žilina region), *Lucka* (Trnava region), *Mitická* and *Tesco* (Trenčín region) and *Evian* from France the low contents of sodium and potassium were determined.The analyses of 8 samples taken from subsurface water, where 4 samples were from Nitra town and its district, another 3 samples were taken from the vicinity of Vrbovce municipality and one sample from the border area with Czech republic showed following composition of all selected elements: the contents of calcium were in range 102.40 – 193.7 mg/L, the contents of magnesium were in range 10.10 – 46.30 mg/L, the contents of sodium were in range 4.10 – 37.50 mg/L and the contents of potassium 0.50 – 18.10 mg/L . In the samples of bottled waters found in stores the data for descriptive characterization were processed with the parametres of functions quantifying the mutual correlation. Strong correlation dependance between the calcium and magnesium contents that could be described by linear linear and square function was identified in analysed waters. The presence of mid strong existency correlation defined by square and non-linear function was evaluated by contents of calcium and sodium in mentioned waters by mathematical program.

Keywords: macroelements, bottled waters, correlation

INTRODUCTION

Macro elements have multiple roles within the body. They work together with vitamins and initiate hormone production as well as speeding up the metabolic processes. Macro elements are the natural elements of which the body needs more amount and are more important than any other minerals. Macrominerals such as sodium and potassium are electrolytes and the body uses electrolytes to maintain acid-base balance and fluid balance (homeostasis) and for normal neurological, myocardial, nerve, and muscle function. Neurons and muscles are activated by electrolyte activity occurring between the extracellular (or interstitial fluid) and intracellular fluid **(Siddiqui, Bawazeer, Joy, 2014)**.
Mineral bottled water, as a safe source of drinking water, has gained a universal popularity in the last few decades **(Cidu, Frau, F. and Tore, 2011)**. Mineral water is the most common type of bottled water **(Doria, 2006)**, which contains essential element cations and anions derived from lithological sources **(Biddau et al., 2019)**. Due to the fact that mineral water has its origin from groundwater resources, the influence of regional and in particular aquifer geology on their chemical composition is expected **(Brenčič, Ferjan, Gosar, 2010; Platikanov et al., 2013)**. Chemical composition of groundwater reflects the local geology of spring source area and the geochemistry of the rocks in contact with the water **(Daniele et al., 2019)**. Groundwater in aquifers may interact with various minerals and become enriched or depleted in some elements **(Tashakor and Modabberi, 2020; Peh, Šorša, Halamić, 2010)**.
Bottled water contains cations such as sodium (Na), calcium (Ca), magnesium (Mg), and potassium (K), and anions such as sulfate (SO_4), chloride (Cl), and bicarbonate (HCO_3). They have good quality due to the special characteristics of the taste and content of various mineral compounds **(Sozoa et al., 2021)**. Generally, bottled water should have a high concentration of Ca and Mg and a low concentration of Na **(Garzon and Eisenberg, 1998)**. Some of the minerals in bottled water, including Ca, Cl^-, Cu, F, I, Fe, Mg, Mn, P, K, Se, Na, Zn, etc., are among the nutrients necessary for the human body **(Gharibzahedi and Jafari, 2017)**. Elevated concentrations of these minerals can reduce the quality of bottled water and be harmful to human health **(Askari, 2021)**.
Groundwater is found as aquifers in underground geologic formations with various lithologies. It flows out naturally as springs, and it can also be pumped from wells. Since water is able to dissolve certain amounts of elements and to transport them for considerable distances, the water that comes to contact with geologic formations is being enriched in chemical elements that are characteristic to those formations. The excess amounts of trace elements may also occur in groundwater as a result of human activities **(Soroush, Ehya, Maleki, 2016)**.
The aim of the work was not only to determine the amounts of exact elements in waters, but also to investigate correlation relations among the contents of selected elements and indirectly analyse by this way the geochemical composition of the rocks which have been influencing surveyed waters.

MATERIAL AND METHODS

Twenty samples of bottled water were randomly chosen from store chain and the data about the amounts of the macroelements (Na, K, Ca, Mg) in mentioned waters were gained from their bottle labels, or this information was taken from web sites. Taking samples of groundwater was carried out with in a pre-washed 0.5 litre plastic bottle (twice with deionized distilled water, with diluted nitric acid, finally again with twice distilled deionized water) from 8 sampling sites - before pouring into these flasks the water samples were filtered through the cellulose membrane Millipore with pores size 0.45 µm. Four sampling sites were situated directly in Nitra town, or in its vicinity, other three in cadastre of Vrbovce by Myjava and one sample was taken from border area with Czech Republic. The samples were analysed with using of atomic absorption spectrophotometer (AAS).
From each 0.5 litre of water 150 ml of water was taken away and preserved by adding of 0.25 ml of conc. HNO_3 to stabilize the metals and then placed the samples to be stored in fridge (by 4 °C) for 3 weeks. The final analysis was carried out with using of Varian AA240FS (Varian, Australia) with 1% HNO_3 as blank.

Then 20 microlitres of the samples were injected into the graphite tube. Concentration of metals were evaluated from established standard curve. All the chemicals used were analytically pure. For the preparation of calibration solutions ultra-purified water was used from the system of purifying of water (BioSan 21001, LabAqua HPLC, Lambda Life) and pure 60 % nitric acid (Merck, Germany).
Parameters for analyses carried out for determinations of macroelements in samples:

K - detection limit - 0.001 mg/L, sensitivity - 0.01 mg/L,
Na - detection limit - 0.04 mg/L, sensitivity - 0.01 mg/L
Ca - detection limit - 0.001 mg/L, sensitivity - 0.01 mg/L
Mg - detection limit - 0.05 mg/L, sensitivity - 0.01 mg/L.

Data processing from determination was carried out in application MS Excel which was also used for calculation of descriptive data value for individual selected samples. The differences in contents of analysed macroelements were verified by the conformity tests of variability of mid-values. By applied methods of regression and corelation analysis in MS Excel the parameters of functions of quantifying and verifying the existence of corelation in occurrence of evaluated amounts of the elements were estimated.

RESULTS AND DISCUSSION

Slovakia with the number, abundance and natural chemical composition of spring waters, mineral and medicinal waters belong to the most important countries in European Union and in the world. Slovak republic is situated from the geomorphological point of view on the territory by **Bodiš et al. (2020)** which is the part of Alpine–Himalayan orogenic belt. As for the ordering of territorial units, all of the bottled waters were originally from the area of Slovakia and were taken from the sources belonging to the Carpathian subsystem (except of bottled waters *Lucka and Tesco* which are from areas belonging to the Pannonian basin subsystem).
Chemical changes of subsurface waters are the results of complex processes taking place in the water – minerals – atmosphere structure. Such physical, chemical and biochemical processes run parallel or one after another. The highest content of calcium (in range 309 - 438 mg/L) were detected in bottled waters *Gemerka* (from Banská Bystrica region), *Salvator, Kaufland Salvatori and Baldovská* (from Prešov region), similarly the highest contents of magnesium (125 – 173 mg/L) were detected in mentioned bottled waters, except for *Baldovská* (graph 1, 2).
The differences have been affected by different types of origin: spring of *Gemerka* is situated in area of Lučensko-košice depression, bottled water *Salvator* from Podhale-Magura area and bottled water *Baldovce* from Fatran-Tatra area.
Content values of sodium (graph 3) were in range 245 - 545 mg/L in bottled waters *Fatra, Budiš and Salvator* (first two springs are situated in Žilina region and mineral water *Salvator* in Prešov region). Mineral waters *Budiš* with *Salvator* and *Kaufland Salvatori* had the highest content of potassium (more than 22 mg/L) (graph 4). Mineral water *Budiš* has come from Fatran-Tatra area and mineral waters *Salvator and Kaufland Salvatori* from Podhale-magura area.
In contrary, the lowest contents of macroelements in interest in bottled waters were as following: the calcium content in range 20 - 47 mg/L were determined in *Fatra and Oravská* (Žilina region) and bottled water *Aro* from Czech Republic (graph 1), content of magnesium in range 2.7-19.5 mg/L was determined by *Rajec, Oravská* (Žilina region) and *Aro* (Czech Republic) (graph 2). The lowest content values of sodium (less than 6.5 mg/L) were detected in bottled waters *Oravská, Rajec, Zlatá studňa from Jasenovo* (Žilina region) *Drobček* (Prešov region), *Lucka* (Trnava region), *Tesco* (Lúka) (Trenčín region), *Aro* (Czech Republic) and *Evian* (France) (graph 3). Bottled waters *Rajec, Zlatá studňa from Jasenovo, Lucka, Tesco and Evian* had the lowest content value for potassium (less than 1.2 mg/L) (graph 4).
Bottled waters *Fatra, Rajec, Zlatá studňa Jasenovo, Tesco* are taken from areas which belong to Fatran -Tatra area from the geomorphological point of view. Spring *Lucka* is situated on the border of Fatran -Tatra area and Záhorská Lowland. Bottled waters *Oravská and Drobček* have their sources at Podhale-magura area. Relatively low presence of tested macroelements were evaluated by two waters from foreign sources: *Aro* (Czech Republic) and *Evian* (France).
Except for mentioned water *Aro* having its origin in abroad, with low content of macroelements, it was obvious also by Slovak bottled waters - *Oravská* bottled water had very low content of all analysed biogenic elements and similarly also bottled water *Rajec* had low contents of other elements, except for calcium.
In table 1 the contents of macroelements in samples of spring waters from taken samples of regions in Slovak Republic are shown. First four samplings were carried out in town Nitra and its vicinity (belonging from the geomorphological division to the area of Podunajská Lowland) and other four samplings were carried out by Vrbovce village in Myjava region (from the geomorphological point of view in area of Slovak-Moravian Carpathians). The contents of calcium were found out in range 102.40 – 193.7 mg/L. The lowest calcium content was determined in sample of groundwater from border area from spring Bílá Studna and on the contrary, its highest content was detected in water from village Podhorany by Nitra. The contents of magnesium were in range from 10.10 to 46.30 mg/L. The lowest amount of Mg was evaluated by the sample from Vrbovce, part Štefanová (no. 1) and its maximal value was evaluated in Nitra (part of Zobor Hill, spring Buganka).
The last two investigated amounts of elements included the contents of sodium in the interval 4.10 – 37.50 mg/L and of potassium 0.50 –18.10 mg/L. Both extremes in contents of lastly mentioned contents were in samples from locality Vrbovce, part Štefanová.
Rapant et al. (2017) referred about the influence of calcium and magnesium in groundwater on the health of inhabitants of Slovak Republic. Their study included evaluation of some more chemical elements in groundwater from Slovakia, also in relation to health of inhabitants. In their works the authors surveyed contents of elements in 49 water samples in two districts of Slovak Republic: Krupina and Bardejov.
Waters from Krupina vicinity had following average contents of macroelements: the sodium content was 13.12 mg/L, calcium content 42.02 mg/L ang magnesium content 12.96 mg/L and analysed waters from Bardejov district had an average sodium content 10.34 mg/L, calcium 80.75 mg/L and magnesium 17.87 mg/L. **Rapant et al. (2017)** concluded that these values are mildly under limit and that health of the inhabitants of Slovak Republic is significantly influenced, also with the life expectancy, by the contents of Ca, Mg and by the hardness of water in groundwaters.
Deteriorated health status and low life expectancy is observed by low deficit content of the parameters of interest in groundwaters. Authors suggest to increase the intake of these cations in drinking water from public sources in following concentrations: Ca > 50 mg /L, Mg > 25 mg/L a Ca+Mg > 2 mmol/L. The team of Michalszki et al. (2018) partly agreed and stated that calcium and magnesium are important components of waters. Authors analysed available bottled waters from the stores in Poland. They evaluated contents of cations, anions and pH. In their studies 48 types of bottled waters were investigated, as following: 25 non-carbonated, 12 mildly carbonated and 11 carbonated waters.
The volume of the bottle was usually 0.5 or 1.5 dm^3, but they analysed also 0.33 and 2.5 dm^3 bottles. Their analyses showed that the middle value of sodium ions in non-carbonated waters was lower than 20 mg/L. In other samples this value varied from 50 to 270 mg/L. Its content did not exceed 10 mg/L in most samples. The highest value of magnesium referred by authors was 131.14 mg/L and of potassium was 31.68 mg/L. The amounts of calcium contents in bottled waters did not exceed according to authors the value 100 mg/L. Güler and Alpaslan (2009) were interested in the concentrations of ions in 70 types of bottled waters from Turkey. The calcium content was in range 0 - 50.9 mg/L, magnesium content was in range 0.1 - 19.0 mg/L, sodium content was detected from 0.1 to 76.8 mg/L and the potassium content was, as authors referred, in range from 0.1 to 76.8 mg/L. Analyses in Slovenian bottled waters available at stores from two years (2004 and 2008) were carried out by Brenčič, Ferjan, Gosar (2010) and their results were published as middle values of these analysed macroelements: content value of calcium was 65.6 mg/L (in 2004) and 76.4 mg/L (in 2008), content of potassium was 1.45 mg/L (2004) and 0.9 mg/L (2008), content of magnesium was 32.4 mg/L (2004) and 34.9 mg/L (2008) and the content of sodium was 9.3 mg/L (2004) and 4.0 mg/L (2008).
Oyebog et al. (2012) also carried out the analyses of the elements present in eight types of bottled waters which were purchased at Camerun market in Middle Africa. Analysed contents of cations in these tested waters were as following: calcium content was in range 0.67 – 57.9 mg/L (with median 19.15 mg/L), magnesium content in range 0.32 – 18.54 mg/L (with median 11.86 mg/L), potassium content was in range 0.19 – 9.18 mg/L (with median 4,46 mg/L) and sodium content was in range 1.45 – 43.94 mg/L (with median 10.64 mg/L). Varrica, Tamburo, Dongarra (2013) carried out the analyses of Sicilian brands of bottled waters. Ranges of mass concentrations of macroelements were as following: 5.39 - 85.3 mg/L as calcium content, 2.67 - 24.9 mg/L as magnesium content, 0.8 - 8.38 mg/L as potassium content and 5.7 - 41.9 mg/L as sodium content. **Felipe-Sotelo et al. (2015)** carried out chemical analyses of bottled waters v in 2014. Their team tested 37 samples of bottled waters from Great Britain and different parts of Europe – Czech Republic, Slovakia, Germany, France, Italy, Finland and Scotland. Authors found out that the bottled waters from Great Britain and Scotland contained lower amounts of macroelements when compared to bottled waters from other countries. **Reyes-Toscano et al. (2020)** analysed the samples of drinking water in Mexico from two samplings, in May and in November in 2018. Contents of calcium were determined in range from 43.74 to 118.02 mg/L in May and from 33.60 to 70.40 mg/L in November. Sodium was the second most abundant element. Sodium contents were in May in range from 17.71 to 52.44 mg/L and in November from 22.08 to 48.30 mg/L. Authors claimed that the tested waters had lower quantities of magnesium in May from 5.47 to 13.62 mg/L and they increased in November and were in range from 18.00 to 35.02 mg /L.
Verifying of correlation relations in the contents of determined macroelements with regression analysis (with using of program application MS Excel) had relevance to parameters of functions affecting the occurrence of individual pairs among elements in bottled waters and in analysed subsurface waters (tab. 2). In this way the geochemical composition of subsoil, in vicinity of particular tested waters occurrence, was indirectly analysed.
In bottled waters purchased in stores the strong corelation between the calcium and magnesium contents could be described with using of linear function where the coefficient of correlation (0.9289) identified the strong relation of dependence (tab. 2) and also the power function where the value of corelation coefficient had the value 0.8979 - on the basis of this findings we could conclude the strong correlation

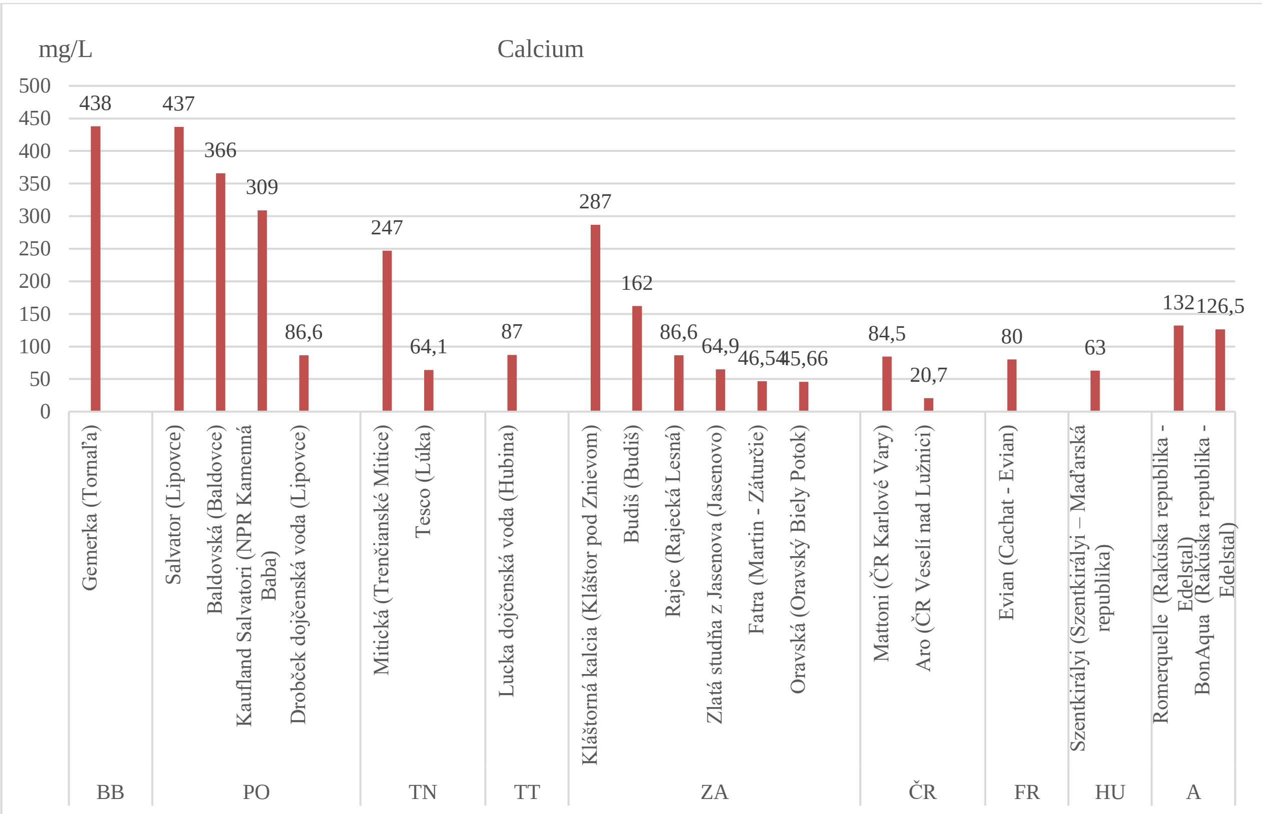
in the contents of both elements in samples of bottled waters from stores in Slovak Republic. Similarly, we can state (based on the results of the regression analysis Software MS Excel) that there is a moderately strong correlation between the calcium and sodium contents, which can be described (tab. 2) using a power function with a correlation coefficient of 0.5550 or a non-linear function (0.5474). From the gained relations we can assume that geological subsoil which is in contact with waters taken from greater depths and purchased in stores is formed by carbonates with predominance of dolomitic subsoil. Dolomite is a mineral with sedimentary origin formed by setting of $CaMg(CO_3)_2$ in hypersaline aquatic environment. It often contains mixtures of clay-like materials (hydrated aluminosilicates with ordered molecules having cations K, Mg, Na, Fe and others). Correlation in contents of sodium and potassium in analysed subsurface waters was evaluated which could be quantified with power function with its parameters which are shown in Table 3. For the other element contents, no significance was confirmed based on the MS Excel outputs. As already mentioned, the samples of these waters come from sources located at low depths, and this could probably be one of the reasons for these detected correlations between the investigated macroelements.

Table 1 Contents of macroelements in samples of subsurface waters and their maximum permissible quantities according to SR Government Regulation 355/2007 Coll.

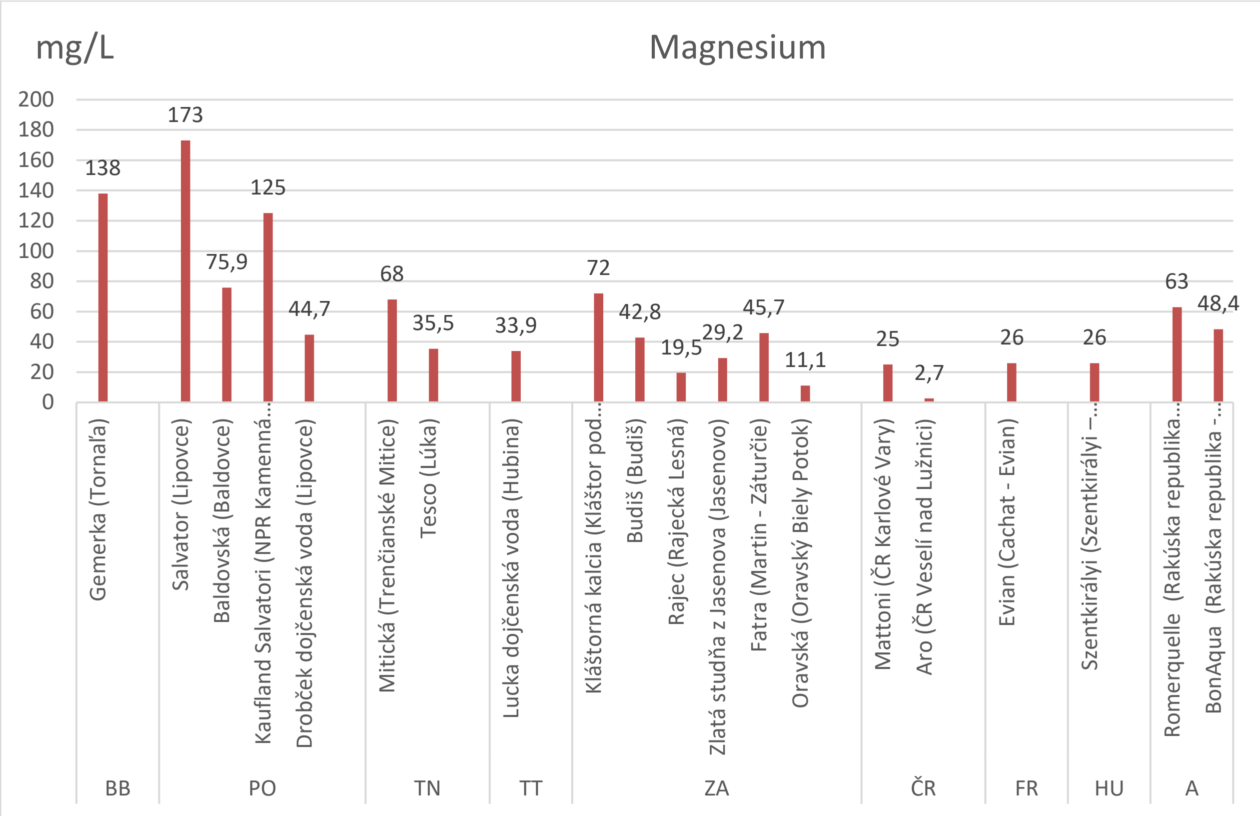
Number of sample	**Ca^{2+} (mg/L)**	**Mg^{2+} (mg/L)**	**Na^{+} (mg/L)**	**K^{+} (mg/L)**	**Water source (Locality of sampling)**
1	193.7	17.70	6.60	6.70	Nitra, Podhorany
2	172.70	21.30	19.40	2.30	Nitra, Šindolka
3	154.30	46.30	34.00	2.70	Nitra, Zobor (Buganka)
4	114.30	32.70	21.70	0.70	Nitra, Mojmírovce
5	130.90	10.10	4.10	0.50	Vrbovce, Štefanová (č.1)
6	143.40	15.80	37.50	18.10	Vrbovce, Štefanová (č.2)
7	155.30	11.60	5.40	0.60	Water source Vrbovce
8	102.40	12.70	4.20	0.06	Bílá Studna
355/2007 Coll.	**-**	**125**	**200**	**-**	

Table 2 Correlation relations between the contents of macroelements in bottled waters

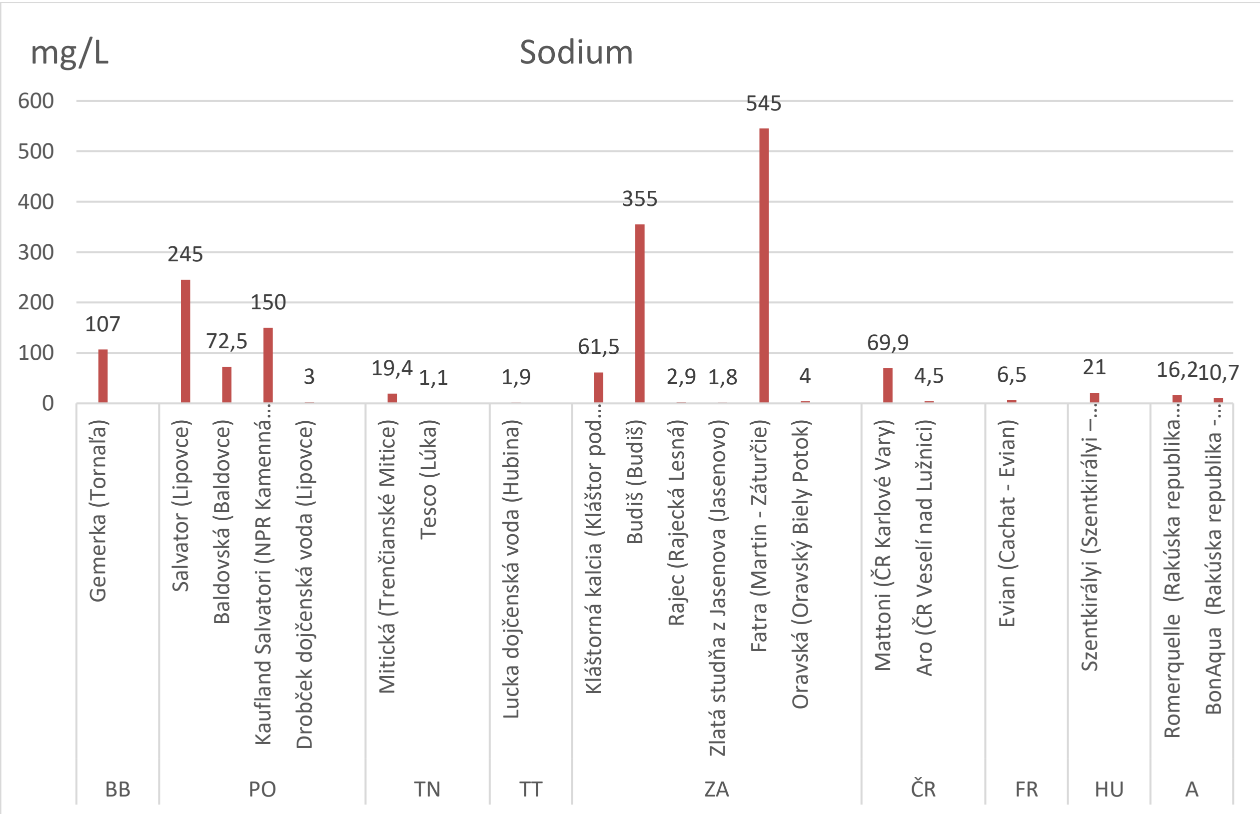
Bottled waters			
Corelation Ca – Mg			
Corelation of equation	*R-square*	*p-value*	*Significance F*
Linear: $y = 2.79x + 6.49$	0.8536	$6.17.10^{-9}$	0.0000
Power: $y = 5.6687x^{0.8178}$	0.8062	$7.88.10^{-8}$	0.0000
Corelation Ca – Na			
Corelation of equation	*R-square*	*p-value*	*Significance F*
Non-linear: $y = -1 : (336.75x) + 214.91$	0.2997	0.0124	0.0124
Power: $y = 55.01x^{0.2468}$	0.3080	0.0111	0.0111



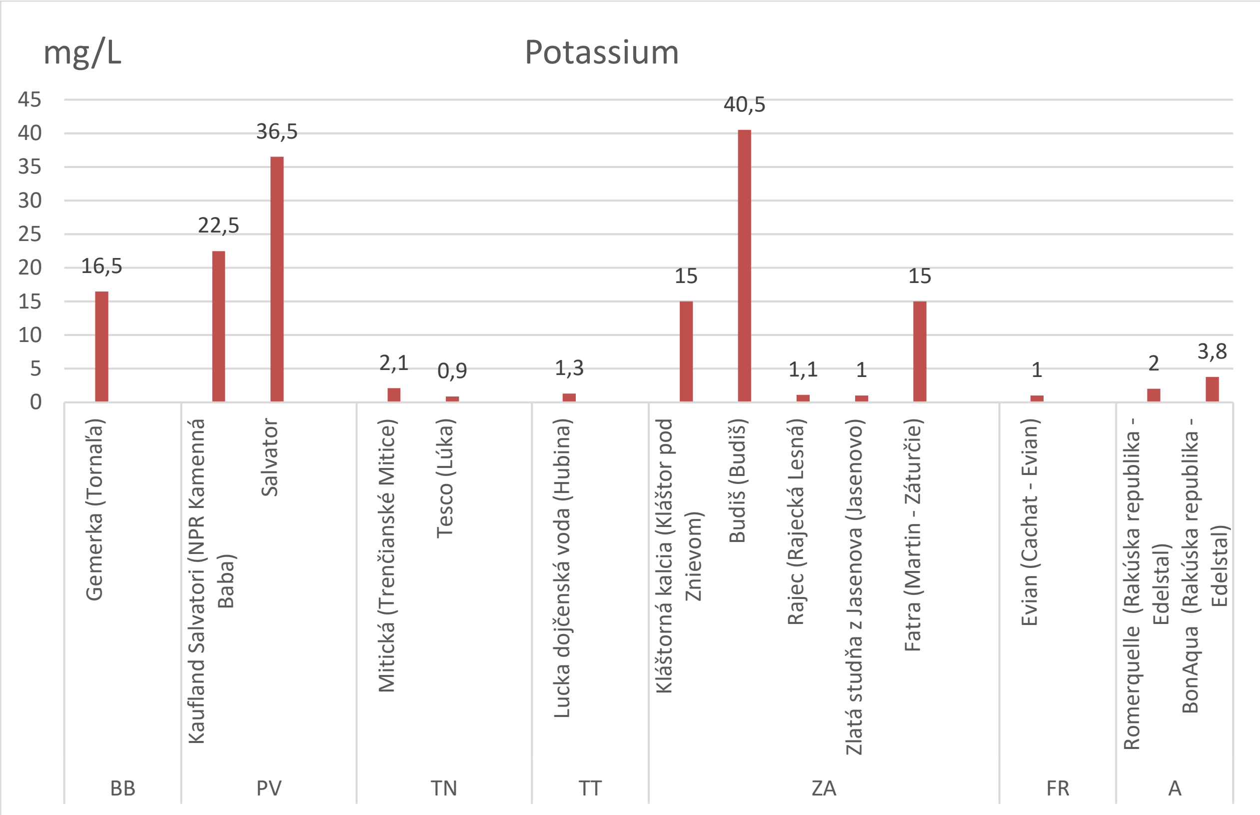
Graph 1 Contents of calcium in bottled waters from five regions of Slovakia and from foreign sources (abbreviations of regions: BB- Banská Bystrica region, PO-Prešov region, TN-Trenčín region, TT-Trnava region, ZA-Žilina region and abbreviations of the states: ČR-Czech Republic, FR- France, HU-Hungary, A- Austria)



Graph 2 Contents of magnesium in bottled waters from five regions of Slovakia and from foreign sources (abbreviations of regions: BB- Banská Bystrica region, PO-Prešov region, TN-Trenčín region, TT-Trnava region, ZA-Žilina region and abbreviations of the states: ČR-Czech Republic, FR- France, HU-Hungary, A- Austria)



Graph 3 Contents of sodium in bottled waters from five regions of Slovakia and from foreign sources (abbreviations of regions: BB- Banská Bystrica region, PO-Prešov region, TN-Trenčín region, TT-Trnava region, ZA-Žilina region and abbreviations of the states: ČR-Czech Republic, FR- France, HU-Hungary, A- Austria)



Graph 4 Contents of pottasium in bottled waters from five regions of Slovakia and from foreign sources (abbreviations of regions: BB- Banská Bystrica region, PO-Prešov region, TN-Trenčín region, TT-Trnava region, ZA-Žilina region and abbreviations of the states: ČR-Czech Republic, FR- France, HU-Hungary, A- Austria)

Sodium and potassium are included among alkali metals in Periodic table and have higher solubility than alkaline earth metals, therefore there is assumption of their mutual corelation in waters affected mainly by precipitation, by which these elements can easily get from surface soils into subsurface waters. It is generally known that drinking water has a concentration of calcium usually 2 to 5 times higher compared to the concentration of magnesium. The average values were found in bottled waters from the stores for calcium 161.71 mg/L and magnesium 55.27 mg/L, that is 2.9-times difference of mentioned averages. The average value of sodium in bottled waters was 84.95 mg/L and of potassium 11.37 mg/L. The average value of calcium in all samples taken for tests was 145.88 mg/L and of magnesium 21.03 mg/L, that is 6.9-times difference of gained averages. The average value of sodium in subsurface waters was 16.61 mg/L and potassium 3.96 mg/L. It was confirmed that our samples taken from the springs of subsurface waters had large proportion of water with its origin from precipitation.
Based on the comparison of the average contents of the considered macroelements in bottled and subsurface waters, it can be concluded that the magnesium content in subsurface springs is highly significantly lower than in mineral waters and the content of sodium and potassium is significantly lower than in bottled waters purchased in stores (tab. 4).

Table 3 Corelation of relations between the contents of macroelements in groundwater

Groundwater			
Corelation Na - Mg			
Corelation of equation	*R-square*	*p-value*	*Significance F*
Power: $y = 6.5432x^{0.4228}$	0.5724	0.0298	0.0298

Table 4 Comparison of average contents of tested macroelements in bottled, as well as in groundwaters

	bottled waters	**groundwater**	**p-value**	**Significance**
Ca	161.71	145.88	0.354	-
Mg	55.27	21.03	0.002	**
Na	84.95	16.61	0.024	*
K	11.37	3.96	0.048	*

Legend: significance: ** is at significance level $p<0.01$ and significance * is at significance level $p<0.05$

CONCLUSION

Macro elements are the natural elements of which the body needs more amount and are more important than any other minerals. Water that comes to contact with geologic formations is being enriched in chemical elements that are characteristic to those formations. Macroelements are the natural elements of which the body needs more amount and are more important than any other minerals. Water that comes to contact with geologic formations is being enriched in chemical elements that are characteristic to those formations. From the determined values, it can be seen that it is not possible to clearly resolve from which regions or geomorphological areas of the Slovak Republic, the waters with the greatest or lowest content of mineral elements originate. It could be stated that relatively low sodium contents are characteristic of *Rajec, Zlatá studňa Jasenovo, Tesco and Lucka* bottled waters, which are obtained from sources in the Fatran-Tatra region. As for the division of water sources from the regions, three bottled waters obtained from the Prešov region (*Salvator, Kaufland Salvatori, Baldovská*) and one water from the Banskobystrický region (*Gemerka*) have relatively high calcium and magnesium contents. The amounts of sodium differed significantly from each other (more than 81-times) in two bottled waters (*Salvator and Drobček*), which have their source in the cadastre of the same city of Lipovce.
By verifying the existence of correlation relationships in the contents of the investigated macroelements, a strong correlation in the contents of calcium and magnesium and a moderate correlation of calcium with sodium in samples of bottled waters sold in stores in the Slovak Republic were detected through regression analysis using the MS Excel application. From these data, it can be concluded that the geological subsoil, which is in close contact with waters taken from greater depths, consists of carbonates with a predominance of dolomite bedrock containing clay-like materials, where K, Mg, Na cations occur.
A moderate correlation between sodium and potassium contents was identified in groundwater. The waters of interest have their springs in lower depths. The assumption of the occurrence of a correlation between these elements in the given waters is mainly justified by the influence of the precipitation activity, by means of which these alkaline metals reach from the surface places of the soil into the investigated waters.

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