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Evaluating the stability of iodine in bottled mineral waters

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ABSTRACT

The paper presents the results of a study aimed at fortifying mineral waters with iodine ions. In the study, natural mineral water with low CO_2 content was used, which had a high content of bioelements and minerals including calcium and magnesium and also included iodine concentrate and lemon flavour. Within the framework of the experiment, several water samples were prepared (1.5 L in volume each), which contained iodine concentrate (0.080 mg/L) and/or lemon flavour (0.5 mL). Additionally, analyses of control samples prepared using distilled water were carried out. In order to identify the changes in the composition of the analysed samples that were fortified with iodine, tests were performed immediately after the preparation of the samples and after 1 h, 4.5 h and 24 h. The results of the study showed a decrease in iodine content by 40% after 24 h. It was noted, however, that the decline in the content of iodides was 10% more rapid in samples of distilled water fortified with iodine than in samples of mineral water. Lemon flavour has a slight impact on the content of iodine determined, although these differences are in most cases statistically insignificant.

1. Introduction

Iodine plays an essential role in human metabolism and in the early development of most organs, including the brain (WHO, 2011). Iodine (atomic number 53) with an atomic weight of 126.9 is the heaviest stable member in group 17 of the periodic table (the halogens). In common with the other stable halogens (fluorine, chlorine and bromine), iodine forms a diatomic molecule, I_2. However, in contrast to the other halogens, elemental iodine exists as a solid which is volatile at room temperature. By gaining an electron to become I^-, iodine acquires an inert element structure. However, the first electron affinity of iodine being relatively low at −295 kJ/mol, I^- loses the acquired electron fairly easily being converted to I_2 (Fuge and Johnson, 2015).

Iodine is an essential element in the human diet and a deficiency can lead to a number of health outcomes collectively termed iodine deficiency disorders (IDD) (Fuge and Johnson, 2015). The daily requirement for this element in humans is approximately 150 μg (Knoch, 1991; Kabata-Pendias and Pendias, 1999; Kabata-Pendias and Szteke, 2012). Iodine deficiency may cause thyroid diseases (goitre symptoms and the related impairment of various metabolic functions – Selinus et al., 2005; WHO, 2011). In deficiency zones, iodine concentration in water typically ranges from 0.1 to 3 μg/L (Kabata-Pendias and Pendias, 1999; Witczak et al., 2013). Ensuring adequate iodine intake is important, particularly among women of reproductive age, because iodine is necessary for early life development. Biologically based dose–response modelling of the relationships among iodide status, perchlorate dose, and thyroid hormone production in pregnant women has indicated that iodide intake has a profound effect on the likelihood that exposure to goitrogens will produce hypothyroxinemia (Lewandowski et al., 2015).

Excess iodine concentrations are also harmful to humans, causing hyperthyroidism and a number of other adverse changes (Selinus et al., 2005). In large doses, far above its normal levels in waters, iodine can be toxic.

As a result of the dominant oceanic source, iodine is strongly enriched in near-coastal soils, but it has been suggested that inhalation represents a very minor source of iodine intake for humans and that even in coastal areas where atmospheric iodine is likely to be elevated, inhalation would provide only an estimated 5 mg/day (Risher and Keith, 2009). Human intake of iodine is mainly from food with some populations also obtaining appreciable quantities of iodine from drinking water (Fuge and Johnson, 2015).

Seafood provides major iodine-rich dietary items, but other inputs are mainly from adventitious sources, such as the use of iodised salt and from dairy produce, which is a rich source mainly due to cattle-feed being fortified with iodine, and to the use of iodine-containing sterilants in the dairy industry. High dietary salt is considered to be the cause of about 30% of hypertension cases among US adults (National Academy of Sciences, NAS, 2010). Globally, approximately one quarter of the adult population has hypertension, a leading risk factor for premature death. High salt intake is also linked to other diseases, including gastric cancer, obesity, kidney stones, and osteoporosis (Lewandowski et al., 2015).

Water is generally an insignificant source of dietary iodine; however, in some circumstances it can be a very major source (Risher and Keith,

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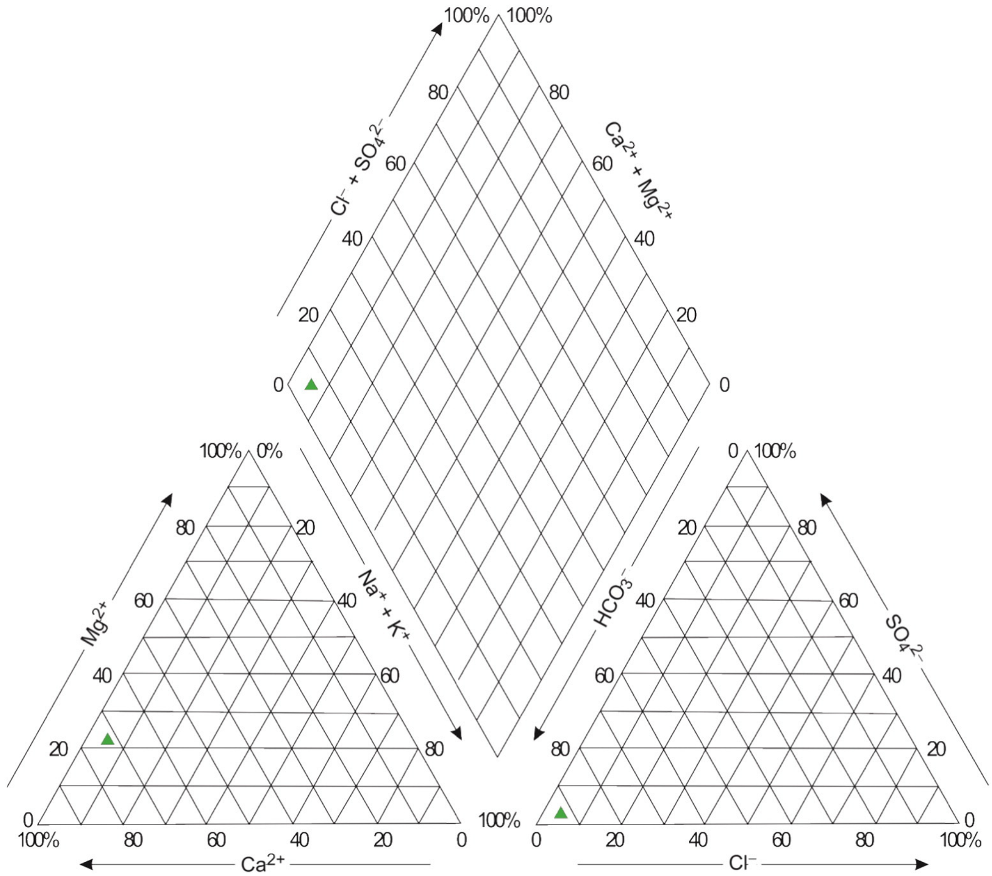

Fig. 1. Piper diagram of analysed water.

2009).Iodine concentrations in groundwater with low mineral content are generally very low – ranging from trace to 60 μg/L (Macioszczyk, 1987). Dojlido (1987) states that the range of iodide concentrations is from 4 to 13 μg/L for groundwater and from 1 to 20 μg/L for surface water. Polish, WHO, EU and U.S. regulations do not set permissible levels for iodine in drinking water (Witczak et al., 2013).

Iodine contents of up to 430 μg/L have been found to occur in the La Pampa Aquifer, Argentina (Smedley et al., 2002; Watts et al., 2010). According to Frengstad et al. (2010) bottled waters available for sale in the Nordic countries (22 products), which originate from Norway, Sweden, Finland and Iceland, contain iodide ions in concentrations ranging from 0.364 to 431 μg/L (with a median of 3.335 μg/L). On the other hand, tap water (18 measurements) had an iodine content ranging from 0.611 to 19 μg/L (with a median of 1.3 μg/L). Iodine concentrations of up to 1220 μg/L have been recorded in water from aquifers hosted by marine sediments in eastern Denmark (Voutchkova et al., 2014). Iodine-rich groundwaters have been found in several provinces of China (Wen et al., 2013), with iodine contents ranging up to 2800 μg/L in Jiangsu Province (Zhao et al., 2000) and up to 4100 μg/L in Shanxi Province (Tang et al., 2013). About half of the groundwater samples, either shallow or deep, contain >150 μg/L (Wen et al., 2013).

Lewandowski et al. (2015) suggested that the human health risks from supplementing drinking water with iodine are negligible; therefore, this approach is worthy of regulatory consideration. Thus it appears reasonable to consider the use of mineral water fortified with iodine as a source of easily assimilated iodine. Mineral water is characterised by its purity at source, its content in minerals, trace elements and other constituents, its storage and its medicinal properties (Petraccia et al., 2006; Ciężkowski et al., 2010). Mineral water can be put on the market and/or exploited for healing purposes (Petraccia et al., 2006).

As the trend towards a healthy lifestyle becomes increasingly popular, many mineral water brands are marketed in developed countries. The first functional water brands that are fortified with selected trace elements, vitamins, herbal extracts or probiotics have also begun to emerge. A significant development of this industry in world markets was observed from 2002 to 2008, mainly as a result of the scale of production and sales of such products in the United States and in Asia. Pioneers in the production and sale of functional water included the Coca-Cola and Pepsi-Cola brands, which were the first to introduce these new products to the U.S. market (ABC Consulting, 2013). In Eastern Europe, this sector is still developing. With growing public awareness of the role of healthy foods and, as a consequence, of the effects of consuming carbonated soft drinks, interest in the consumption of bottled table and mineral water has increased. In Poland, an increase in the sales of these products was observed particularly in the first years of the 21st century.

Table 1
Physical and chemical properties of mineral water tested (the results of the water quality provided by the producer).

Parameter	Concentration [mg/L]
TDS	1341.50
Na^+	17.55
K^+	3.35
Ca^{2+}	233.00
Mg^{2+}	44.08
Cl^-	17.30
SO_4^{2-}	24.60
HCO_3^-	961.3
I^-	<0.01
CO_2	500

Table 2
Results of determination of iodide ions in the samples analysed. Explanation: N – normal sample, D – duplicate sample.

Sample code	Sample description	True value [mg/L]	Results [mg/L]			
			Immediately after preparation	After 1 h	After 4,5 h	After 24 h
T1	Distillate water + iodine	0.080	0.098	0.075	0.080	0.054
T1s	Distillate water + iodine (shaked)		0.102	0.080	0.083	0.055
T2	Distillate water + iodine + lemon flavour		0.098	0.081	0.079	0.058
T2s	Distillate water + iodine + lemon flavour (shaked)		0.096	0.082	0.079	0.059
T3	Mineral water + iodine (N)		0.070	0.061	0.068	0.045
T3s	Mineral water + iodine (shaked) (N)		0.065	0.058	0.060	0.042
T4	Mineral water + iodine (D)		0.062	0.053	0.058	0.041
T4s	Mineral water + iodine (D) (shaked)		0.061	0.053	0.054	0.040
T5	Mineral water + iodine + lemon flavour (N)		0.063	0.052	0.057	0.042
T5s	Mineral water + iodine + lemon flavour (shaked) (N)		0.063	0.054	0.056	0.041
T6	Mineral water + iodine + lemon flavour (D)		0.062	0.056	0.057	0.040
T6s	Mineral water + iodine + lemon flavour (D) (shaked)		0.061	0.055	0.056	0.040

Currently the market has stabilised and consumers are looking for new healthy natural products. Their expectations could in the future be met by natural functional waters based on mineral waters.

In the Polish functional water market, the following products are offered, inter alia:

- Zbyszko brand waters: Veroni Mineral Fit in six flavours: 1) lemon + vitamin C; 2) strawberry + calcium; 3) apple + zinc; 4) peach + fiber; 5) grapefruit + magnesium; 6) coconut + L-carnitine (ABC Consulting, 2013);
- water with collagen by Voda Naturalna Sp. z o.o. (www.vodanaturalna.pl);
- Jupik Aqua Sport water fortified with vitamins B12, B9 and B3 by Hoop Polska Sp. z o.o. (www.hoop.com.pl);
- Ustronianka Sp. z o.o. brand waters: 1) with iodine; 2) with magnesium; 3) with calcium; 4) with potassium (www.ustronianka.com.pl/4-ustronianka_funkcjonalna.html).

The first waters fortified with iodine on the Polish market were launched in 2009 by Ustronianka Sp. z o.o. The product is based on natural mineral water. It is water with a mineral content of approx. 507 mg/L and with low sodium content (6.4–7.1 mg Na/L), containing calcium (90–117 mg/L), magnesium (16.5–21.3 mg/L), potassium (approx. 1 mg/L), carbohydrates (295–299 mg/L), sulphates (41.8–48.8 mg/L) and chlorides (21.1–26.7 mg/L) (Mineral and spring waters test, 2011). It has been registered as a foodstuff intended for a particular nutritional use recommended for pregnant women and lactating mothers, and also for those who require a low-sodium or sodium-free diet. One litre of this product contains 150 μg of iodine, which is 100% of the recommended dietary allowance (RDA) (Product Cart).

Water and functional beverages are included in the “functional food” category, i.e. food products with documented beneficial effects on human health resulting from the presence of nutrients considered essential (Gawęcki, 2002). The European Union recognises the definition established under the FUFOSE (Functional Food Science in Europe) European research programme, according to which “foods can be regarded as functional if they can be demonstrated to affect beneficially one or more target functions in the body, beyond nutritional effects, in a way relevant to an improved state of health and well-being and/or reduction of risk of disease” (Kudełka, 2011).

The term “functional water” is understood as bottled mineral, spring or purified water with added vitamins, minerals, botanical extracts including herbs, probiotics, ozone and functional components (e.g. mallow, coenzyme Q10, Omega-3 fatty acids and oxygen). Functional ingredients may also act as flavouring agents (Petraccia et al., 2006; Barroso et al., 2009; Peacock et al., 2013). Functional water is generally clear. Juices (below 10%) may also be added to it – Nestlé Wellness is an example here. According to the current state of our knowledge and research, functional water producers typically use ready additives (food products) in their technological processes: vitamins, carotenoids and other technological additives for the food, pharmaceutical, cosmetics and fodder industries (Dege (2011); ABC Consulting, 2013; Peacock et al., 2013).

This paper presents the results of tests concerning the stability of iodine concentrations in mineral waters fortified with this element. It is assumed that mineral water will be fortified with iodine in an amount covering half the daily requirement of this element for the human body. Additional studies were conducted for water that included lemon flavour in addition to iodine. The results of these tests may be relevant for entrepreneurs who wish to produce functional water, i.e. bottled mineral water fortified with iodine.

2. Materials and methods

2.1. Calculation of the amount of iodine added to mineral water

According to WHO (2011), the daily requirement of iodine for humans is 0.15 mg. Since iodine is volatile and is easily released from

Table 3
t-Test. Comparison of the results obtained for samples with and without the addition of lemon flavour.

Paired samples	Paired differences					t[a]	df[b]	Significance (2-tailed) p
	Mean	Standard deviation	Standard error mean	95% confidence interval of the difference				
				Lower	Upper			
T1–T2	−0.0030	0.002915	0.001304	−0.006620	0.000620	−2.301	4	0.083
T1s–T2s	0.0010	0.004123	0.001844	−0.004120	0.006120	0.542	4	0.616
T3–T4	0.0084	0.003578	0.001600	0.003958	0.012842	5.250	4	0.006
T3s–T4s	0.0034	0.001949	0.000872	0.000980	0.005820	3.900	4	0.018
T5–T6	0.0002	0.001924	0.000860	−0.002188	0.002588	0.232	4	0.828
T5s–T6s	−0.0008	0.001095	0.000490	−0.002160	0.000560	−1.633	4	0.178

[a] The t statistic is obtained by dividing the mean difference by its standard error. Results below zero indicate that mean iodine concentration in samples without lemon flavour is higher than mean iodine concentration in samples with lemon flavour and vice versa.
[b] Degrees of freedom.

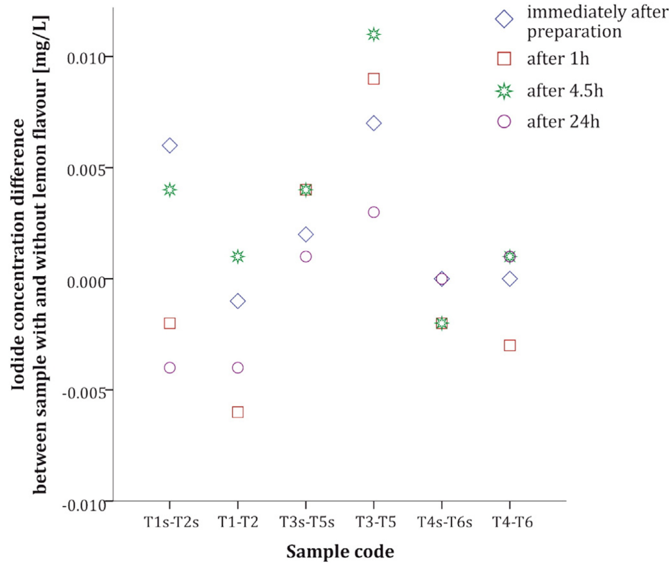

Fig. 2. Differences between the results obtained for samples with added iodine concentrate and the results obtained for samples with added iodine concentrate and lemon flavour.

water, a slightly higher concentration of 0.08 mg rather than 0.075 mg was adopted in order to prepare samples with a concentration of iodide ions corresponding to half the daily requirement of this element for the human body. For this purpose 0.0725 mL of iodine concentrate with a concentration of iodide ions 1650 mg/L and a density 1.198 mg/mL was added to 1.5 L of mineral water. This concentration corresponds to concentrate mass 0.087 g.

In the study, potassium iodide concentrate with organogenic origin (produced from the decomposition of marine algae) was used.

2.2. Test procedure

Natural mineral water with low CO_2 content was used in testing. It had a high content of bioelements and minerals including calcium and magnesium. It is bicarbonate-calcium-magnesium (HCO_3-Ca-Mg) mineral water (Fig. 1).

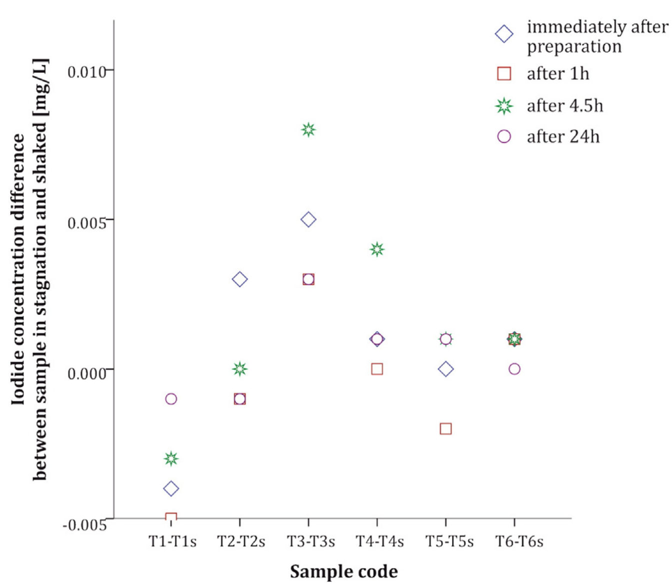

Fig. 3. Differences between the results obtained for stagnant and agitated samples.

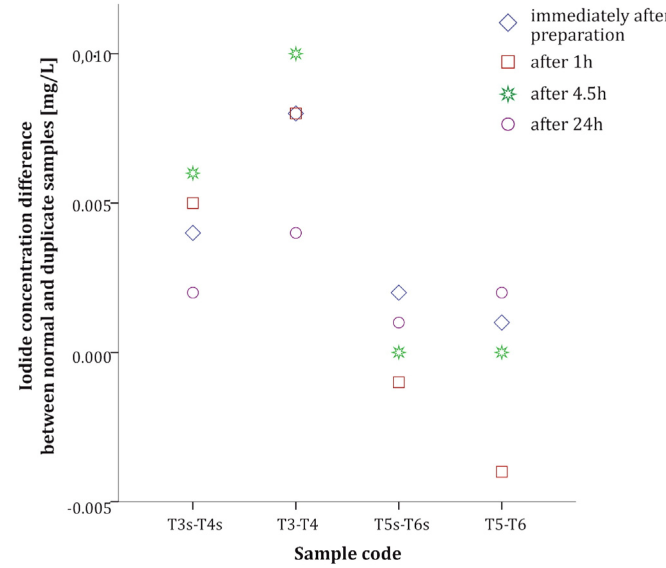

Fig. 4. Differences between the results obtained for normal and duplicate samples.

The physical and chemical properties of the water tested are shown in Table 1.

Within the framework of the experiment, several water samples were prepared (1.5 L in volume each), which contained iodine concentrate and/or lemon flavour (0.5 mL):

1. mineral water + iodine concentrate (T3 - sample code from Table 2);
2. mineral water + iodine concentrate + lemon flavour (T5);
3. mineral water + iodine concentrate (analysis after the bottle was vigorously agitated) (T3 s);
4. mineral water + iodine concentrate + lemon flavour (analysis after the bottle was vigorously agitated) (T5 s).

In order to control the results obtained, the aforementioned tests (1–4) were conducted on two parallel sets of samples, normal samples (N) and duplicate samples (D):

T3-T4, T3s-T4s, T5-T6, T5s-T6s (see Table 2).

Additionally, analyses of control samples prepared from distilled water were carried out:

1. distilled water + iodine concentrate (T1);
2. distilled water + iodine concentrate + lemon flavour (T2);

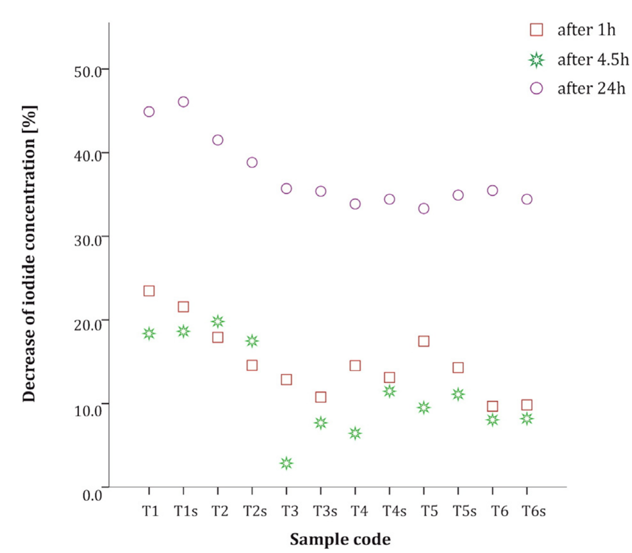

Fig. 5. Percentage decrease in iodide ion concentrations in time.

Table 4
Percentage decrease in iodide ion concentrations in time for distilled and mineral water.

Water type (sample codes)	Decrease after 1 h [%]	Decrease after 4.5 h [%]	Decrease after 24 h [%]
Distillate water with iodine (T1, T1s)	21.6–23.5	18.4–18.6	44.9–46.1
Distillate water with iodine and lemon flavour (T2, T2s)	14.6–17.9	17.5–19.8	38.8–41.5
Mineral water with iodine (T3, T3s, T4, T4s)	12.8–14.5	2.8–11.5	33.9–35.7
Mineral water with iodine and lemon flavour (T5, T5s, T6, T6s)	9.7–17.5	9.5–11.1	33.3–35.5

3. distilled water + iodine concentrate (analysis after the bottle was vigorously agitated) (T1 s);
4. distilled water + iodine concentrate + lemon flavour (analysis after the bottle was vigorously agitated) (T2s).

The tests were conducted for polyethylene (PE) bottles.

In order to identify the changes in the composition of the analysed samples fortified with iodine in time, tests were performed immediately after the preparation of the samples, and then after 1 h, 4.5 h and 24 h.

Chemical analyses were performed at the accredited Hydrogeochemical Laboratory of the Department of Hydrogeology and Engineering Geology, Faculty of Geology, Geophysics and Environmental Protection of the AGH University of Science and Technology in Kraków (accreditation certificate PCA No. AB 1050).

Iodide concentration was determined using inductively coupled plasma mass spectrometry (ICP-MS) according to the PB-01 ed. 3 test procedure of 4 May 2010 based on the PN-EN ISO 17294-2 (2006) standard. The limit of quantification of this method for iodide ions is DL = 0.01 mg/L. Precision as expressed by the relative standard deviation (RSD) does not exceed 10%, while accuracy determined on the basis of recovery (R) varies from 92% to 111%, depending on concentration. At the concentration level of 0.08 mg/L precision is around 5%, and recovery is equal to 95%. Total uncertainty of the determination of iodide ion concentration at this concentration level is 13%. This means that for a concentration of this element in the sample at approx. 0080 mg/L, values in the range from 0.070 to 0.090 mg/L lie within the uncertainty boundaries for the test.

2.3. The statistical method

In order to verify the results of investigations especially whether differences obtained are statistically significant, the t test for dependent samples was performed using the PS Imago Academic (http://psimago.pl/), based on IBM SPSS Statistics, licensed to AGH.

The test (also called the paired t-test or paired-samples t-test) can determine whether the difference between the mean concentrations of iodine in dependent sample sets is statistically significant, i.e. whether we can reject the null hypothesis (which assumes that the means do not differ) at a set level of significance (0.05) (Warner, 2008). If the significance of the test (p-value) is less than 0.05 ($p < 0.05$), we reject the null hypothesis that assumes that the means are equal for both sample sets and we assume that the difference between the means is statistically significant. If the significance of the test (p-value) is greater than 0.05, we have no reason to reject the null hypothesis that the mean concentrations of iodine in two sets of samples are equal. Full example of the test results is presented only for the samples containing flavour and samples without flavour (see Table 3).

3. Results and discussion

The results of experiments are summarised in Table 2.

Based on the analysis results obtained, iodine concentrations in all measurement series were compared. The analysis of the results obtained clearly shows that with time, the concentration of iodide ions decreases and after 24 h it is lower approximately by half.

Differences between the results obtained for samples with added iodine concentrate and the results obtained for the same samples with added iodine concentrate and lemon flavour are shown in Fig. 2.

On the basis of the relationships shown in Fig. 2, it can be concluded that the results obtained for samples without the addition of lemon flavour are higher than for those with the flavour added.

In order to verify whether these differences are statistically significant, the t test for dependent samples was performed (Table 3). The purpose of the test is to verify whether iodine determination results for the samples containing flavour are different from those for the samples without flavour. The null hypothesis (H_0) is that iodine determination results for the samples containing flavour do not differ from those for the samples without flavour.

According to the data shown in Table 3, the differences are statistically insignificant (significance of the test, p-value, is greater than 0.05), so we have no reason to reject the null hypothesis that the average concentrations of iodine in samples with and without flavour are equal. For normal samples T3–T4, T3s–T4s the differences are statistically significant (significance of the test, p-value, is lower than 0.05). They may result from the fact that the addition of lemon flavour causes water to become “effervescent”, which promotes the release of volatile iodine compounds and a reduction in the concentration of this element).

It was also analysed whether the results of iodine determinations for samples that were agitated differ from the results obtained for unagitated samples (Fig. 3).

The analysis showed that the results of iodine determination for unagitated samples were slightly higher than those for agitated ones. These differences are negligible and are not statistically significant (t-test significance $p > 0.05$ with the exception of T1–T1s, T3–T3s samples). Additionally, the presence of several results where the difference is zero or where the figures for agitated samples are indeed higher than for unagitated ones suggest that these differences are due to errors occurring during sample preparation and the analysis itself. However, these results are within the range of measurement uncertainty.

The results of iodine determination for normal and duplicate samples were compared as well (Fig. 4).

The analysis conducted showed that the results obtained for normal samples were slightly higher than those for duplicate samples (Fig. 4), but the differences were very small and amounted to less than 10% of the value obtained, although in two cases (samples T3–T4, T3s–T4s) they were statistically significant (significance of the test, p-value, is lower than 0.05).

Fig. 5 show the percentage decrease in iodide ion concentrations for subsequent measurement series.

The analysis of the results obtained clearly demonstrates that the concentration of iodide ions decreases with time. After 24 h, the value was lower by about 40%.

It has been noted, however, that the decline in the content of iodides was 10% more rapid in samples of distilled water fortified with iodine than in mineral water samples (Table 4), which should be considered favourable.

Table 4. Percentage decrease in iodide ion concentrations in time for distilled and mineral water.

4. Conclusions

The results of the tests conducted for bicarbonate-calcium-magnesium (HCO_3-Ca-Mg) mineral water fortified with iodine lead to the following conclusions:

1. Minor differences in the results of tests carried out for individual samples result from low iodine concentrations in the water tested. This variation is associated with the low precision and high uncertainty of iodide ion determinations at concentrations that are close to the laboratory limit of quantification.
2. Lemon flavour has a slight impact on the content of iodine determined, although these differences are in most cases statistically insignificant.
3. Agitating the water bottle does not affect the results of determinations and consequently the concentration of the iodine dissolved in the water.
4. The concentration of iodine in the water decreases over time and therefore the product should have a shelf life of 24 h after opening.
5. The release of iodine from mineralised water is approx. 10% slower compared to its release from distilled water.
6. Owing to the rate of release of iodine from water (more than 10 % after just one hour), it is recommended that the concentration of iodine added to mineral water be increased to 0.15 mg/L, which is in line with WHO (2011) with regard to the human body's daily requirement of iodine.
7. Mineral water fortified with iodine may prove an important market segment aimed at enriching the diet with magnesium and calcium and also with iodine, which is highly beneficial to health. In this context, the results of research obtained may be relevant for entrepreneurs who wish to produce functional water, i.e. bottled mineral water fortified with iodine.

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