



Chemical composition of organically and conventionally grown fruits of raspberry (*Rubus idaeus* L.) cv. *Willamette*

Mira Milinković^{1*}, Dragana Vranić¹, Milena Đurić², Svetlana M. Paunović¹

¹Fruit Research Institute Čačak, Kralja Petra I 9, 32000 Čačak, Serbia

²Faculty of Agronomy, University of Kragujevac, Cara Dušana 34, 32000 Čačak, Serbia

*Corresponding author: miramilinkovic@yahoo.com

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ABSTRACT

The paper presents two-year results of chemical tests of raspberry fruits in organic and conventional cultivation systems. Raspberry plantations were established on the slopes of Jelica Mountain, on soil having very acidic reaction (pH/KCl 3.67–3.76) and optimal contents of humus and total N. In the conventional cultivation system, the soil had a high supply of readily available P₂O₅ and K₂O and microelements, with excessive levels of Ni, and Cr. In the organic system, the content of Ni and Zn increased. The biochemical properties of organic and conventional fruits showed no significant differences, while the influence of the research year and the interaction of the cultivation system showed significant differences in the content of TA and IS. However, higher values of the levels of soluble solids, total acids and sugars contributed to the more appealing taste and aroma of organic raspberry fruits. The contents of microelements and heavy metals in the fruits of both cultivation systems were measured in the following order Mn>Fe>Zn>Cu>Ni>Cr>Co. No significant difference was found in the contents of the examined elements between organic and conventional fruits, except for the statistically higher content of Fe in organic raspberries. The high share of individual elements (especially Ni) in both cultivation systems indicates the need for continuous chemical testing of soil and fruits. Based on the results, it is important to consider the daily intake of each element in relation to body weight and/or maximum daily intake.

Keywords: organic production, raspberry, chemical composition of fruits, microelements.

ИЗВОД

У раду су приказани двогодишњи резултати хемијских испитивања плодова малине у органском и конвенционалном систему гајења. Засади малине засновани су на обронцима планине Јелице, на земљишту јако киселе реакције (pH/KCl 3,67–3,76) и оптималног садржаја хумуса и укупног N. У конвенционалном систему гајења, земљиште је високо обезбеђено лакоприступачним P₂O₅, K₂O и микроелементима, са прекомерним садржајем Ni и Cr, а у органском систему гајења, повећан је садржај Ni и Zn. Биохемијске особине плодова органског и конвенционалног система гајења не показују значајне разлике, док утицај године истраживања и интеракцијски односи система гајења показују значајне разлике у садржају TA и IS. Ипак, више вредности садржаја растворљиве суве материје, укупних киселина и шећера утичу на пријемчивији укус и арому органских плодова малине. Садржај микроелемената и тешких метала у плодовима оба система гајења измерен је према следећем редоследу Mn>Fe>Zn>Cu>Ni>Cr>Co. Утврђено је да не постоји значајна разлика у садржају испитиваних елемената у плодовима органског и конвенционалног система гајења, осим статистички вишег садржаја Fe у органским плодовима малине. Висок удео појединих елемената (посебно Ni) у оба система гајења указује на потребу континуираног хемијског испитивања земљишта и плодова, а на основу резултата је неопходно сагледати дневни унос појединачног елемента у односу на телесну тежину и/или максималан дневни унос.

Кључне речи: органска производња, малина, хемијски састав плода, микроелементи.

1. Introduction

Using health-safe food produced without the application of pesticides, synthetic fertilizers and growth biostimulants has been a global tendency. Organic farming is a system that improves and promotes the health of ecosystems, including agroecosystems, specifically focusing on the preservation of biodiversity, biological cycles and soil biological activity, by biological, mechanical and agricultural methods, excluding the use of synthetic

materials (FAO, 1999; Golijan and Veličković, 2015; Popović et al., 2016).

Organic production requires knowledge and monitoring of all agroecological factors and anthropogenic impacts in the field of growing. Fruits may contain significant amounts of toxic elements or heavy metals as a result of atmospheric deposition, urban and industrial activities and agronomic practices. Agronomic practices are the main source of heavy metals in agricultural soils (Silva et al., 2005; Willett, 2002; He et al., 2005). The quality of organic products is the foundation for high food demand, and thus

stimulates the intensive development of organic production (Jovanović et al., 2017). There is considerable worldwide increase in agricultural area under organic cultivation from 50.9 million ha in 2015 to 11 million ha in 1999 (Willer and Lernoud, 2017). Consumer demand for red raspberries (*Rubus idaeus* L.) is due to their pleasant taste and nutritional quality as well as potent health promoting benefits (Krishkova and Tsoleva, 2020), which indicates the need to monitor these parameters in the soil under raspberry plantations and their impact on fruit quality. The total world production of raspberries in 2016 was 795 249 tonnes. The primacy of Europe in the entire world production can primarily be seen in the fact that six countries from this continent are among the top ten world producers of raspberries, including Serbia (Grčak et al., 2019). Organic raspberry production in Serbia has been increasing, and the first plantations based on organic production methods were registered in 1999 (Mišić et al., 2004).

Based on the above, the aim of the research was to determine the chemical characteristics of raspberry soil and fruits under organic and conventional growing systems.

2. Materials and methods

2.1. Plant material and cultural practices

Tests were performed in raspberry cv. *Willamette* plantations under organic and conventional production systems in 2018–2019. Raspberry plantations under both types of production were established in 2015, on the slopes of Mountain Jelica in the village of Grab (Municipality of Lučani).

Under organic growing system, cultural practices included the application of aged manure in the amount of 30 t ha⁻¹ in 2017 and the organic fertilizer Italpolina (NPK 4:4:4) at 1,000 kg ha⁻¹ before the beginning of the growing season in 2018 and half of the stated rate in 2019. Copper and sulfur-based agents were used for plant protection.

In the conventionally grown raspberry plantation, the fertilizers NPK 6:12:24 in the amount of 500 kg ha⁻¹ and KAN (27% N) in the amount of 300 kg ha⁻¹ were

applied in both years of research. Disease and plant management was carried out according to the standard protection program.

Fruits were selected visually and were at the same stage of development during sampling. They were taken from similar portions of the bush. During intensive harvesting, when most of the fruits were at full maturity (first half of July), fruits were harvested for chemical analysis and the soil was sampled in order to determine the contents of microelements and heavy metals. All analyses were performed in the Laboratory for Chemical Testing of the Fruit Research Institute, Čačak.

2.2. Agromechanical and agrochemical characteristics of soil

The mechanical composition of the soil was determined by the sieving and sedimentation method – (JSSS, 1997). The proportion of physical sand fractions was 39.90–46.70%. The soil under raspberry plantation was clay loam with a low proportion of clay fractions and a high proportion of physical clay (55.30–60.10%). The texture of the raspberry orchard soil under organic and conventional systems is shown in Table 1.

The agrochemical analyses of the soil included the following testing: pH in 1N KCl and pH in H₂O (potentiometrically); CaCO₃ content (Scheibler calcimeter), humus content (Kotzman), nitrogen (Kjeldahl), readily available phosphorus and potassium (AL method, P₂O₅ – colorimetry and K₂O – flame photometry).

In both raspberry production systems, the average values of active soil acidity (pH/H₂O) were in the acid range (4.57–4.75), and exchangeable acidity had strongly acid values (3.67–3.76 pH/KCl). The contents of humus and total nitrogen were higher in organic raspberry plantations (4.85% and 0.24%, respectively), and the levels of readily available phosphorus (28.24 mg 100g⁻¹ air-dry soil) and potassium (38.90 mg 100g⁻¹ ads) were higher than in conventional production. The values of basic soil fertility parameters are shown in Table 2.

Table 1.
Soil texture (%)

Growing system	Depth (cm)	Proportion of particle size fractions (%)					
		CS 2–0.2	FS 0.2–0.02	S 0.2–0.02	C <0.002	PS >0.02	PC <0.02
mm							
Organic	0–30 cm	19.05	27.65	34.20	19.10	46.70	55.30
Conventional	0–30 cm	11.03	28.87	43.60	16.50	39.90	60.10

CS – Coarse sand; FS – Fine sand; S – Silt; C – Clay; PS – Physical sand; PC – Physical clay

Table 2.
Values of basic soil fertility parameters

Growing system	Year	pH		CaCO ₃	Humus (%)	Total N	P ₂ O ₅ mg/100g ads	K ₂ O
		H ₂ O	KCl					
Organic	2018	4.88	3.67	0.20	5.07	0.25	20.39	36.30
	2019	4.61	3.84	0.28	4.63	0.23	15.98	35.60
average		4.75	3.76	0.28	4.85	0.24	18.19	35.95
Conventional	2018	4.48	3.49	0.28	4.72	0.24	32.52	42.20
	2019	4.66	3.85	0.42	4.04	0.20	23.95	35.60
average		4.57	3.67	0.35	4.38	0.22	28.24	38.90

2.3. Contents of microelements and heavy metals (Zn, Cu, Fe, Mn, Co, Ni, Cr) in soil

The total concentrations of microelements and heavy metals in soil samples were determined by the extraction method (HCl/HNO₃, 3/1) (McGrath and Cunliffe 1985) and measured using a PinAAcle 500 Perkin-Elmer atomic absorption spectrophotometer (USA 2018).

2.4. Chemical analyses of raspberry fruit

Raspberry fruit samples were collected at the stage of full maturity in three replicates, from each variant of the experiment, and then used to test the chemical composition.

2.4.1. Biochemical analyses of fruits

The soluble solids content (SSC) of the fruit was determined by a manual refractometer (Hanna Instruments, Germany). The dry matter content was determined by drying at 105°C until constant mass was reached. Titratable acidity (TA) was determined by neutralizing fruit extract with 0.1 N NaOH to pH 8.2, using phenolphthalein as indicator. Acidity was expressed as mg citric acid 100 g⁻¹ fresh weight. Sucrose, inverted sugars, and total sugars were determined by the Luff-Schoorl method (Tanner and Brunner, 1979).

2.4.2. Contents of microelements and heavy metals (Zn, Cu, Fe, Mn, Co, Ni, Cr) in raspberry fruits

The contents of microelements and heavy metals were analyzed using the modified method (Moraisa et

al., 2017). Readings were performed on an AAS, Perkin-Elmer, PinAAcle 500 (USA 2018), and values were expressed in mg kg⁻¹ of dry weight of the sample.

2.5. Statistical analysis

Each variant of the experiment was analyzed in three replicates, and the results were presented as mean and standard error. Data were analyzed by one-way analysis of variance (ANOVA) to examine differences among the cultivars, using Statistica 7 (StatSoft, Inc., Tulsa, OK, USA). The pairwise comparisons between different parameters were performed using Duncan's Multiple Range Test, $P < 0.05$.

3. Results and discussions

Plants accumulate metals from the soil during growth, and as metals are not biodegradable, they can enter the food chain, which may lead to increased human intake, causing serious illness (Jabeen et al., 2010). Zinc, copper, iron, chromium and cobalt are essential elements which become toxic only at high concentrations (Radivojevic et al., 1999). It is thus important to monitor metals in soil, fruit and fruit products.

Based on the results in Table 3, statistically significantly higher contents of Cu, Fe, Mn, Co and Cr were obtained under the conventional growing system than under the organic system. Zn content was higher in the organic system (123.1 mg kg⁻¹) compared to the conventional (64.9 mg kg⁻¹). Differences between sampling years were found for Co and Cr contents. The interaction of the cultivation systems were significant for all examined elements, except for Mn.

Table 3.

Contents of total microelements and heavy metals in soil (mg kg⁻¹ air dry soil)

Factor		Zn	Cu	Fe	Mn	Co	Ni	Cr
Growing system (A)	Organic	123.1 ± 5.8 a	32.6 ± 1.7 b	26477.2 ± 1525.9 b	1577.5 ± 75.3 a	25.9 ± 1.1 b	99.5 ± 8.06 a	65.1 ± 2.9 b
	Conventional	64.9 ± 2.6 b	39.9 ± 2.1 a	34639.5 ± 1590.3 a	1860.8 ± 135.0 a	30.7 ± 3.8 a	102.7 ± 6.4 a	84.8 ± 9.2 a
Year (B)	2018	96.3 ± 16.5 a	37.4 ± 2.9 a	30658.7 ± 2503.9 a	1703.3 ± 109.7 a	32.6 ± 2.8 a	99.6 ± 7.61 a	86.5 ± 8.3 a
	2019	91.7 ± 10.3 a	35.1 ± 1.8 a	30458.7 ± 2290.4 a	1735.0 ± 140.7 a	24.1 ± 1.6 b	102.7 ± 6.9 a	63.3 ± 3.0 b
AxB	Organic 2018	132.0 ± 8.9 a	31.7 ± 2.8 b	25704.7 ± 2369.7 c	1465.0 ± 29.9 a	26.6 ± 0.7 b	82.9 ± 1.9 b	68.5 ± 2.9 b
	Organic 2019	114.2 ± 8.9 b	33.4 ± 2.3 b	27249.7 ± 2330.4 bc	1690.0 ± 121.6 a	25.2 ± 2.3 b	116.2 ± 6.6 a	61.7 ± 4.8 b
	Conventional 2018	60.7 ± 2.6 c	43.0 ± 2.3 a	35612.7 ± 1091.7 a	1941.7 ± 49.8 a	38.6 ± 1.8 a	116.3 ± 2.6 a	104.5 ± 3.1 a
	Conventional 2019	69.2 ± 3.1 c	36.9 ± 2.7 ab	33666.3 ± 3241.3 ab	1780.0 ± 286.6 a	22.9 ± 2.5 b	89.2 ± 4.2 b	65.0 ± 4.5 b
ANOVA								
A		**	*	*	ns	*	ns	*
B		ns	ns	ns	ns	*	ns	**
AxB		*	*	*	ns	*	*	*

Values with different letters denote statistically significant differences (Duncan's Multiple Range Test, $P < 0.05$)

Legal regulations have not specifically regulated the limit values of heavy metals in the soils used for organic production. Based on the Regulations on Permitted Quantities of Hazardous and Deleterious Substances in Soil and Irrigation Water and Their Testing Methods No. 23/1994, the content of Ni in both

growing systems had increased values 82.9–116.2 mg kg⁻¹ (maximum allowable concentration – mac 50 mg kg⁻¹), whereas the content of Cr (104.50 mg kg⁻¹) increased in conventional growing system (mac 100 mg kg⁻¹). Based on the Regulation on Limit Values of Polluting, Deleterious and Dangerous Substances in the

Soil, No. 30/2018 and 64/2019, the content of Cu (mac 36 mg kg⁻¹), Ni (mac 35 mg kg⁻¹) and Co (mac 9 mg kg⁻¹) was increased in both growing systems whereas the content of Cr (100 mg kg⁻¹) increased in the conventional system. Kabata-Pendias and Pendias (2001) set permitted limit values for Co (50 mg kg⁻¹), Cr (100 mg kg⁻¹), Cu (100 mg kg⁻¹), Ni (100 mg kg⁻¹) and Zn (300 mg kg⁻¹).

The availability of heavy metals, as well as their uptake by plants, depends on numerous factors, primarily their total and accessible content in the soil, soil reaction, the content of organic matter, oxidation-reduction conditions, etc. (Adriano, 2001; Kabata-

Pendias, 2011), and the uptake of heavy metals depends on plant species as well (Overesch et al., 2007). Increased Cu content in soil and fruits may result from the use of copper-based products for disease protection.

The quality of the fruit depends on its physicochemical properties and chemical ingredients. Organic acids, especially malic acid and citric acid, contribute to berry fruit acidity. The ratio of sugars to organic acids determines the balance between soluble solids and titratable acidity, which in turn influences fruit flavor and taste (Papaioanou et al., 2018).

Table 4.
Biochemical composition of raspberry fruit

Factor		Soluble solids content SSC (%)	pH values	Titratable acidity TA (%)	Total sugars TS (%)	Invert sugars IS (%)	Sucrose (%)
Growing system (A)	Organic	9.95 ± 0.52 a	3.05 ± 0.51 a	1.99 ± 0.13 a	5.40 ± 0.32 a	3.96 ± 0.31 a	1.37 ± 0.12 a
	Conventional	8.98 ± 0.48 a	3.15 ± 0.21 a	1.78 ± 0.19 a	4.68 ± 0.27 a	3.48 ± 0.43 a	1.14 ± 0.23 a
Year (B)	2018	9.23 ± 0.62 a	3.25 ± 0.52 a	1.63 ± 0.13 b	5.40 ± 0.29 a	4.32 ± 0.23 a	1.03 ± 0.13 a
	2019	9.70 ± 0.43 a	2.96 ± 0.16 a	2.14 ± 0.13 a	4.68 ± 0.31 a	3.12 ± 0.33 b	1.48 ± 0.18 a
AxB	Organic 2018	10.10 ± 0.96 a	3.14 ± 1.09 a	1.82 ± 0.14 ab	5.76 ± 0.40 a	4.44 ± 0.24 a	1.25 ± 0.14 a
	Organic 2019	9.80 ± 0.62 a	2.96 ± 0.32 a	2.15 ± 0.19 a	5.04 ± 0.48 a	3.48 ± 0.42 ab	1.48 ± 0.19 a
	Conventional 2018	8.35 ± 0.50 a	3.35 ± 0.39 a	1.43 ± 0.17 b	5.04 ± 0.35 a	4.20 ± 0.43 a	0.80 ± 0.13 a
	Conventional 2019	9.60 ± 0.73 a	2.95 ± 0.18 a	2.12 ± 0.21 a	4.32 ± 0.34 a	2.76 ± 0.49 b	1.48 ± 0.35 a
ANOVA							
A		ns	ns	ns	ns	ns	ns
B		ns	ns	*	ns	*	ns
AxB		ns	ns	*	ns	*	ns

Values with different letters denote statistically significant differences (Duncan's Multiple Range Test, $P < 0.05$)

The results on the chemical properties of the fruit shown in Table 4 show that there is no difference in the biochemical parameters examined between the organic and conventional cultivation systems. The influence of the research year and the interaction (AxB) of the cultivation systems showed significant differences in the content of TA and IS.

There are no literature data on the biochemical composition of organically cultivated raspberry fruits, while significant research has been conducted in conventional systems. Kulina et al. (2012) determined that, in cv. Willamette, SSC was 10.00%, TS 3.42%, IS 2.55%, sucrose 0.83%, TA 1.35, pH 3.00, and these values were lower than our research values. Raspberry pH ranged from 2.17 to 2.99 (Papanov et al., 2020). Gülçin et al. (2011) found higher values for pH (3.66), SSC (11.66%) and TA (1.11%) compared to our tests. Studies by Miletić et al. (2012) determined higher values for most parameters, except pH and sucrose, in cv. Willamette, compared to our research.

Studies investigating the effect of farming system on the contents of minerals and toxic elements are

uncommon but are important in predicting possible benefits or risks to human health from consuming organic or conventional foods (Magkos et al. 2006). Microelements involved in the proper functioning of enzymes can also have a negative impact on human health, depending on the amount taken into the body. Table 5 shows the results on the content of microelements and deleterious — substances in raspberry fruit.

The research results show that the content of Fe in organic raspberry fruits was statistically significantly higher than in conventional ones, and the influence of the year of examination was especially pronounced (Tab. 5). The influence of the research year on the content of Mn, Ni and Cr was also determined.

The content of other microelements and heavy metals in the fruits did not differ in relation to the cultivation system, regardless of the significant differences from their values in the soil. As regards their content in the fruit, Miclean (2000) reported that Cu in agricultural products should be between 4 and 15 ppm.

Table 5.
Content of microelements and deleterious substances in raspberry fruit (mg kg⁻¹ of the sample dry matter)

Factor		Cu	Zn	Fe	Mn	Ni	Co	Cr
Growing system (A)	Organic	4.52 ± 0.2 a	12.86 ± 0.9 a	20.31 ± 5.1 a	20.90 ± 2.9 a	3.52 ± 0.7 a	0.61 ± 0.1 a	0.94 ± 0.3 a
	Conventional	5.52 ± 0.8 a	13.53 ± 0.8 a	4.73 ± 0.9 b	19.36 ± 1.6 a	3.29 ± 0.6 a	0.94 ± 0.2 a	0.77 ± 0.2 a
Year (B)	2018	4.57 ± 0.2 a	12.49 ± 0.7 a	7.12 ± 1.4 b	16.22 ± 1.3 b	4.78 ± 0.4 a	0.95 ± 0.2 a	1.24 ± 0.2 a
	2019	5.47 ± 0.8 a	13.90 ± 0.9 a	17.32 ± 6.3 a	24.04 ± 1.9 a	2.03 ± 0.2 b	0.60 ± 0.1 a	0.47 ± 0.1 b
AxB	Organic 2018	4.43 ± 0.2 a	12.21 ± 1.0 a	9.54 ± 2.4 b	15.31 ± 2.4 b	4.92 ± 0.8 a	0.63 ± 0.1 b	1.44 ± 0.2 a
	Organic 2019	4.61 ± 0.4 a	13.50 ± 1.7 a	31.07 ± 2.8 b	26.49 ± 2.6 a	2.12 ± 0.4 b	0.59 ± 0.2 b	0.44 ± 0.1 c
	Conventional 2018	4.71 ± 0.3 a	12.77 ± 1.3 a	5.89 ± 0.6 b	17.12 ± 1.2 b	4.63 ± 0.3 a	1.27 ± 0.2 a	1.04 ± 0.3 ab
	Conventional 2019	6.33 ± 1.58 a	14.29 ± 1.11 a	3.57 ± 1.4 a	21.59 ± 2.4 ab	1.94 ± 0.24 b	0.60 ± 0.16 b	0.50 ± 0.07 bc
ANOVA								
A		ns	ns	*	ns	ns	ns	ns
B		ns	ns	*	*	*	ns	*
AxB		ns	ns	*	ns	ns	ns	ns

Values with different letters denote statistically significant differences (Duncan's Multiple Range Test, $P < 0.05$)

However, FAO (Codex Alimentarius Commission, 2011) and other studies recommend daily levels of intake through food, and an intake value of 0.5 mg kg⁻¹ of adult body weight for Cu. The upper limit of the acceptable range of oral intake in adults is uncertain but is most likely several but not many mg/day in adults (several meaning more than 2 or 3 mg day⁻¹). The same directive suggests that the average daily intake of Fe has been estimated to be 17 mg day⁻¹ for males and 9–12 mg day⁻¹ for females, while the recommended intake of Zn is 0.3–1 mg kg⁻¹ of body weight. Human health assessment data revealed that Ni intake should be limited to 0.020 mg kg⁻¹ body weight per day (US-EPA IRIS; Haber et al. 2017). No limit for Cr in foods is given in the Codex Alimentarius Commission, or by Australia, New Zealand, Japan, the United States and Taiwan. Data for Cr in blueberries are given by Hua et al. (2014), who found 0.77 mg kg⁻¹. Zeiner and Juranović-Cindrić (2018) reported the range of 0.01–1.5 mg kg⁻¹ of dry weight for Cr content in the fruits of different plant species, which is in agreement with our results.

The National Health and Nutrition Examination Survey, which provides dietary intake data for most nutrients, does not include manganese. Records on the usual Mn intake dose in healthy population are 1.2–2.2 mg for males and 1.2–16.0 mg for females, for the age 1–18, whereas for the age above 19, 2.3 mg for males and 1.8 mg for females (Institute of Medicine, Food and Nutrition Board, 2001). The same source states that Tolerable Upper Intake Levels (ULs) for Manganese 2.0–9.0 mg up to 18 years and 11.0 mg for older than 19, regardless of sex.

The area of our research has a geological predisposition for the increased presence of Fe, Mn, Ni, Cr and Co, which together with the acid reaction of the soil leads to increased uptake of these elements by plants. No significant difference was found in the contents of the tested elements between the raspberry growing systems, which is in line with the research by Pires et al. (2015), who found no significant difference in the content of microelements in the fruits of mango, persimmon, Barbados cherry and strawberry between organic and conventional growing systems. A similar

conclusion was reached by Forman and Silverstein (2012), who determined that the nutrient composition of organic products did not differ significantly from conventional products. Based on the above, it is necessary to continue comparative studies of the chemical composition of the fruits of organically and conventionally grown plants, in order to determine the amount of daily intake of individual elements, when consuming raspberry fruits. The selection of raspberry growing sites, in addition to planned procedures, should involve a detailed analysis of the agrochemical properties of the soil, including the contents of microelements and heavy metals, with the aim of monitoring metal accumulation in fruits.

4. Conclusions

Organic raspberry fruits have higher contents of soluble solids, acids and sugars, which affect the taste and flavor of the fruit. Significantly higher levels of microelements and heavy metals were determined in conventional soil than in organic production, but it did not affect the difference in their content in fruits from different cultivation systems. The content of Fe was higher in organic fruits. However, the excessive content of Ni, Cr and Co, as well as the high content of Fe and Mn, together with the strong acid reaction of the soil, contributed to the high uptake of these elements by raspberry fruits in both cultivation systems. The consumption of large amounts of tested raspberry fruits can satisfy a significant percentage of the daily intake of these elements. Based on the above, continuous chemical tests are needed in order to determine the amount of intake of an individual element in accordance with the recommended doses per day/body weight and/or age, which are in accordance with the recommendations of the medical profession.

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