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The Influences of Genotype and Year on Some Biologically Active Compounds in Honeysuckle Berries

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Abstract: Berries of three Romanian *Lonicera caerulea* cultivars 'Cera', 'Kami', and 'Loni', grown at the Research Institute for Fruit Growing, Pitesti, Romania, were analyzed between 2020 and 2022 in terms of chemical composition. The study aimed to determine the concentrations of some compounds with antioxidant activity, highlight the most valuable cultivar, encourage the consumption of honeysuckle berries, and indirectly stimulate growers' interest in this little-known species in Romania. Some phenolic compounds—lycopene, β-carotene, and vitamin C—were quantified. As a result of the study, the 'Loni' cultivar's high total phenolic content, flavonoids, anthocyanins, vitamin C, lycopene, chlorogenic and neochlorogenic acids, catechin, and rutin are to be noted. 'Cera' cultivar had the highest cryptochlorogenic acid content, and 'Kami' summarized the highest carotenoid level. These characteristics indicated that the three honeysuckle cultivars' berries could have multiple uses, from fresh consumption, as part of a diet focused on maintaining human health, to being used as raw materials in the para-pharmaceutical industry, to obtain food supplements. The novelty characteristics and the nutritional value of its berries highlighted by this study have indicated that honeysuckle can become a crop of interest and profitability.

Keywords: β-carotene; cultivars; lycopene; *Lonicera caerulea*; phenolic components; vitamin C

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1. Introduction

Lonicera caerulea, a member of the *Caprifoliaceae* family, is originally from the Holarctic temperate zone. It is relatively little known, although it has a history with deep roots and written evidence dating back to the pre-Linnaean period, when Clusius, in 1583, provided a description and a picture of a honeysuckle with blue fruits [1]. This gives the honeysuckle an advantage for those consumers looking for unexperienced pleasurable tastes. Nevertheless, the financial success of a lonicera plantation depends on the market's preferences, and new food's acceptance is linked to the consumers' education, income, taste, and previous experience [2]. The honeysuckle produces ovoid fruits that normally bulge in the center and are narrow at the ends, are dark blue, are covered by a cuticular wax layer, weigh 0.7–1.3 g, and mature in stages [3]. Their berries' sour taste is shaped mainly by the content of organic acids, and among the phenolic compounds, tannins, together with the iridoid glycosides and malic and citric acid esters, are responsible for the bitter sensation [4]. Depending on the cultivar, the fruits can be sweet–sour, sweet–bitter, or sour–bitter. The honeysuckle is favored by its early presence on the market, as honeyberries are the first fruits to ripen, starting from the middle of May, depending on the cultivar and the climatic conditions [3]. Through its composition, the honeysuckle is placed among the most valuable sources of

antioxidants, and the species' great ability to adapt to different ecological conditions and their resistance to the very low temperatures of the winter period [3] reduce the risks of losing much of the fruit yield. In addition, under the conditions of a changing climate only in certain periods during the year and with a predisposition to climatic accidents in the spring [5–7], the productivity and even the survival of some species must be taken into account. To all this is added the fact that the involvement of honeysuckles in culture in different areas gives it the advantage of reaching consumers quickly, fresh, and without additional costs related to long-distance transport. In general, climate (along with pedological factors) is the determining factor in productivity increases or decreases and the quality of fruit agroecosystems. The relationship among the genetic background of fruit species, agricultural practices, and local environmental conditions represents production's quantitative and qualitative basis [8–11]. Plants produce a wide range of secondary metabolites, and among them, phenolic compounds have been progressively synthesized during their evolution. It is appreciated that berries, including honeyberries, have higher contents of bioactive compounds. Many of these compounds have medical or socio-economic value, justifying the interest of the scientific world and the fruit industry [10–15]. *Lonicera caerulea* shows high health potential and is a promising source of numerous bioactive compounds, mainly anthocyanins, phenolic acids, and flavonols [16]. In several studies, phenolic compounds' protection against chronic diseases such as hypertension, diabetes, cardiovascular diseases, and atherosclerosis has been documented. Furthermore, phenolic compounds were shown to have beneficial effects on cognitive processes, ophthalmology conditions, and antibacterial activity, especially in kidney infections [12,13]. Other reported properties of honeyberry fruit involve antimicrobial, anti-inflammatory, anti-atherosclerotic, and anticarcinogenic activities, which have been demonstrated in in vitro and some in vivo tests [4]. *Lonicera caerulea* was introduced in Romania, at the Research Institute for Fruit Growing, Pitesti, Romania, in 1985. Three Romanian cultivars were obtained through free pollination, and two of them ('Loni' and 'Cera') were registered in 2004. The 'Kami' cultivar was registered seven years later, in 2011. Sumedrea et al. [3] described 'Loni' as a cultivar with medium–high vigor, erect and compact growth, and the ability to produce ovoid, dark-blue berries of 0.7–1 g, slightly covered in wax, with a sweet and sour taste. 'Cera' produces fruits of about 0.9 g with an obovate shape. The cultivar has good resistance to frost, drought, diseases, and pests. 'Kami' is a high-vigor cultivar with large fruits (1.0–1.3 g), a pleasant, almond-like taste, and resistance to drought and frost. Although it has very high adaptability to the unfavorable conditions of early spring (late frosts) or summer (dry periods), productivity-related data do not present honeysuckle as a very attractive crop. What could ensure the success of a lonicera plantation are the characteristics related to its berry biochemical composition that open opportunities for its exploitation. Starting from these considerations, this study was carried out over 2020–2022 to highlight the content of some bioactive compounds in berries of the three *Lonicera caerulea* cultivars bred in Romania, i.e., 'Cera', 'Kami', and 'Loni'. Additionally, this paper's concern is to highlight the most valuable Romanian honeysuckle cultivar to encourage this species' cultivation in farms.

2. Material and Method

2.1. Experimental Site

The experiment was carried out between 2020 and 2022 in the *Lonicera caerulea* plantation of the Research Institute for Fruit Growing, Pitesti, Romania (R.I.F.G., 44°51′38″ N 24°52′4″ E, 285 m above sea level) on three Romanian honeysuckle cultivars ('Cera', 'Kami', and 'Loni'). The plant material intended for planting was produced through in vitro micropropagation at the R.I.F.G. The establishment year of the honeysuckle plantation was 1991, the planting distances were 3 × 1.2 m, and the canopy shape was a bush. The soil presented the characteristics of the wet phreatic alluviosol protisols class and has a loam-sandy granulometric composition, with a moderately acidic reaction. The data related to the climatic conditions in the period preceding the ripening of honeysuckle fruits, between

2020 and 2022, were obtained from the WatchDog900ET automated weather station, which is about 400 m from the haskap plantation. These data are presented in Table 1, together with the multiannual values of the same months (from the period 1969–2021).

Table 1. Climatic parameters registered in January–June of 2020–2022 and their multiannual values (1969–2021).

Climatic Parameters		Year	January	February	March	April	May	June
Air temperature °C	Monthly mean	2020	0.3	4.2	7.7	10.9	15.0	19.6
		2021	0.5	3.0	4.1	8.6	15.6	19.3
		2022	0.8	3.1	3.6	10.1	16.4	21.1
		1969–2021	−1.2	0.5	4.9	10.4	15.4	18.9
Sunshine (Sh, monthly sum, hours)		2020	162.1	148.7	171.4	296.6	243.7	266.3
		2021	91.0	145.6	160.3	176.8	266.2	259.9
		2022	161.1	161.4	185.4	215.3	286.0	286.3
		1969–2021	99.9	115.6	160.2	193.9	246.2	276.0
Rain (Monthly sum, mm)		2020	1.8	22.5	30.0	21.1	104.1	166.2
		2021	73.6	12.4	66.8	38.4	65.4	104.0
		2022	6.4	10.8	19.4	88.0	72.6	25.6
		1969–2021	33.9	33.0	37.7	55.0	81.8	100.6

2.2. Sampling

Berries of the honeysuckle cultivars (samples of approximately 200 g per cultivar and harvest) were harvested at full maturity and visually assessed by the appearance of intense violet-blue coloring and the ease of the detachment of the berries from the plant. Three harvests were performed for each genotype, between the last week of May and the first week of June, and the samples were kept at −18 °C until the time of extract preparation (about 14 days).

2.3. Chemicals and Reagents

The following chemicals and reagents were used: methanol, hydrochloric acid, distilled water, hexane, ethanol, acetone, Folin–Ciocalteu reagent (Merck-Sigma-Aldrich, Darmstadt, Germany), sodium carbonate, sodium nitrite, aluminum chloride, sodium hydroxide, potassium chloride, sodium acetate, 2,6-dicholorophenolindophenol sodium salt dihydrate, sodium bicarbonate, phosphoric acid, acetonitrile, and standards: gallic acid monohydrate, catechin hydrate, cyanidin chloride, ascorbic acid, epicatechin, rutin hydrate, quercetin dihydrate, isoquercetin, chlorogenic acid, cryptochlorogenic acid, and neochlorogenic acid (Merck-Sigma-Aldrich, Darmstadt, Germany).

2.4. Analysis Methods

2.4.1. Extraction Procedures

To obtain the methanolic extracts of the honeysuckle berries, 1 g frozen berry homogenate samples were treated with 10 mL of methanolic solution (8:2, v/v) and vortexed for 2 min at 3000 rpm. Afterward, the mixture was subjected to ultrasonic treatment (40 kHz) for 180 min, to extract polyphenols and 150 min, for the extraction of flavonoids. This step was followed by centrifugation for 15 min at 3000 rpm, and the resulting supernatant was filtered and used for analysis. For the HPLC analyses, the obtained methanolic extracts were subjected to evaporation at room temperature, until a constant mass was reached, resulting in a semi-solid consistency product, which was kept at −18 °C until further determination. Before the HPLC analysis, the semi-solid product was dissolved in concentrated methanol (99.8%) by ultrasonication at a temperature below 30 °C and filtered through Macherey–Nagel (MV) filters, with 0.20 µm pores. To obtain the methanolic extract necessary for the determination of monomeric anthocyanins, 1 g of frozen berry homogenate was added to a solution containing about 9 mL of methanol and 1 mL of 27%

hydrochloric acid [17]. The mixture was vortexed for 2 min at 3000 rpm and kept in the dark for 10 min, and then filtered. The filtrate was later used for the determination of monomeric anthocyanins. To obtain the aqueous extracts necessary for total tannin content determination, 1 g of frozen berry homogenate was treated with 10 mL of distilled water and subjected to vortex (2 min, 3000 rpm), followed by ultrasonication (30 min, 80 °C). The mixture was subsequently centrifuged (15 min, 3000 rpm), and the supernatant was used further for analyses. To obtain the extract necessary to determine the vitamin C content, a cold extraction was performed: 1 g of frozen berry homogenate was treated with 10 mL of 1% hydrochloric acid solution and subjected to vortex treatment for 2 min at 3000 rpm. After 10 min in the dark, the solution was filtered and used for determination. All extraction procedures were performed at a low temperature (4 °C, maintained by adding ice) and low light. For carotenoid extracts, a berry sample (1 g frozen berry homogenate) was added to a mixture of 25 mL of solvents (hexane: ethanol: acetone in a volume ratio of 2:1:1). The mixture was stirred for 30 min at 1500 rpm, 10 mL of distilled water was added, and stirring was continued for another 10 min. Afterward, the extraction was carried out by manual stirring every few hours during 72 h of rest in the dark at room temperature.

2.4.2. Determination of Total Phenolic Content

The determination of total content of phenolic compounds (TPC) was carried out by the spectrophotometric method, according to the methodology proposed by Cosmulescu et al. [18]. The principle of the method is based on the formation of a blue compound, as a result of the reaction carried out in an alkaline environment between phospho-tungstic acid and polyphenols. The reaction mixture consisted of 0.5 mL of honeysuckle methanolic extract added to a 10 mL flask containing 7 mL of distilled water and 0.5 mL of Folin-Ciocalteu reagent. After 5 min of resting, 2 mL of 10% sodium carbonate solution was added. After another 60 min, the absorbance of the samples was measured and the concentration of polyphenols was estimated. Gallic acid monohydrate was used as a standard for calibration, and the total polyphenol content was expressed in mg gallic acid equivalent (GAE) 100 g^{-1} fresh weight (FW).

2.4.3. Determination of Total Tannin Content

To determine the total tannin content (TTC) in honeysuckle berries, the methodology proposed by Giura et al. [19] was followed. In a 10 mL flask containing honeysuckle aqueous extract and Folin–Ciocalteu reagent, a 10% sodium carbonate solution was added. Absorbances were read after 60 min at 760 nm wavelength. For calibration, gallic acid monohydrate was used as the standard compound. The total concentration of tannins was calculated and expressed in mg GAE 100 g^{-1} fresh weight (FW).

2.4.4. Determination of Total Flavonoid Content

For the quantification of flavonoids, the methodology proposed by AL-Ghudani and Hossain [20] was used. The principle of the method is based on the formation of a yellow–orange compound through the reaction of flavonoids and aluminum chloride. To the initial reaction mixture, consisting of 1 mL of methanolic extract in a 10 mL volumetric flask containing 4 mL of distilled water and 0.3 mL of 5% sodium nitrite, after 5 min in the absence of light, 0.3 mL of 10% aluminum chloride was added. After standing for 5 min, a 2 mL solution of 1 M sodium hydroxide was added, and the sample was diluted with distilled water to a final volume of 10 mL. The absorbance of the solution was measured at 510 nm. For calibration, catechin hydrate was used as a standard, and the total flavonoid content (TFC) of the samples was expressed in mg catechin equivalent (CE) 100 g^{-1} fresh weight (FW).

2.4.5. Determination of Total Monomeric Anthocyanin Content

To determine the total monomeric anthocyanin content (TAC), a protocol similar to Anggraini et al. [17] was followed. The differential pH method is based on the reversible

change in the color of a solution of monomeric anthocyanins with the change in pH. The difference in absorbance of the pigments at 520 nm is proportional to the concentration. The extracts were diluted with two different buffer solutions, potassium chloride 0.025 M (pH 1.0) and sodium acetate 0.4 M (pH 4.5), and the absorbance was read after 30 min at 520 and 700 nm. To calculate TAC, Anggraini et al.'s [17] formula was used. Cyanidin-3-glucoside chloride was used as a standard, and the results are expressed in equivalents of cyanidin-3-glucoside (C3G) 100 g^{-1} fresh weight (FW).

2.4.6. Determination of Vitamin C Content

To quantify the ascorbic acid content, 10 mL of the extract (obtained according to the previously detailed procedure) was titrated with a 2,6-dichloroindophenol solution, according to Segura Campos et al. [21]. The final titration point was considered to be the appearance of a pink-rose color persisting for more than 5 s. Ascorbic acid was used as a standard, and the results are expressed in mg 100 g^{-1} fresh weight (FW).

2.4.7. Determination of Lycopene and β-Carotene Content

The quantitative determination of carotenoids (lycopene and β-carotene) was carried out following the protocol proposed by Tudor-Radu et al. [22]. The concentrations of two carotenoids in the specific extracts, expressed in mg 100 g^{-1} plant material, were calculated, using molar extinction coefficients of 184,900 M^{-1} cm^{-1} at 470 nm and 172,000 M^{-1} cm^{-1} at 503 nm for lycopene, and 108,427 M^{-1} cm^{-1} at 470 nm and 24,686 M^{-1} cm^{-1} at 503 nm for β-carotene in hexane.

2.4.8. HPLC-DAD Analysis

The SHIMADZU LC-20AT HPLC system equipped with a quaternary pump, solvent degasser, autosampler, and UV-Vis detector with a photodiode (DAD) was used. The separation of the compounds was performed on a 150 × 4.6 nm, 5 µm Kinetex C18 column (Agilent Technologies, Santa Clara, CA, USA), using the gradient mode, for 45 min at a temperature of 35 °C, with a flow rate of 0.8 mL min^{-1}. The injection volume of standards/samples was 10 µL. The mobile phase consisted of a gradient mixture of eluent A (water and phosphoric acid pH 2.3) and eluent B acetonitrile. The components of the mobile phase were filtered through Macherey–Nagel (MV) filters with a0.20 µm pores and degassed in an Elma-Elmasonic P ultrasonic bath to remove air bubbles. The linear gradient elution used was as follows: 0–28 min, 5–50% B; 28–38 min, 50–65% B; 38–40 min, 65–30% B; 40–41 min, 30–5% B; 41–45 min, 5% B. The detection wavelengths were set to 280, 320, and 360 nm. For the quantification of phenolic compounds in the samples, calibration curves were drawn for the concentration ranges: 2.5–35 µg mL^{-1} (catechin, epicatechin, rutin, and quercetin), 3.63–50.75 µg mL^{-1} (chlorogenic, neochlorogenic, and cryptochlorogenic acids), and 4–56 µg mL^{-1} (isoquercetin). The results are expressed in mg 100 g^{-1} or mg 100 g^{-1} chlorogenic acid equivalents (for chlorogenic acid and its isomers; neochlorogenic and cryptochlorogenic acids).

2.5. Statistical Analysis

All analyses were performed in three replicates. The extracts necessary for the determination of TPC, TTC, TFC, TAC, and vitamin C were analyzed in triplicate in each year of the study. For carotenoids and the HPLC analysis (i.e., determination of the levels of chlorogenic, neochlorogenic, and cryptochlorogenic acids; and catechin, rutin, and isoquercetin), three analyzes were carried out, one for each year of study (essentially, the data from the three years of study were considered in this paper as three replicates), only considering the effect of the cultivar, but not the study year effect. Therefore, two-way ANOVA, followed by Duncan's multiple range test ($p < 0.05$), was used to study the cultivar and experimental year's effects on total phenolics, tannins, flavonoids, monomeric anthocyanins, and vitamin C. One-way ANOVA, followed by Duncan's multiple range test was used to study the cul-

tivar's effects on lycopene; β-carotene; chlorogenic, neochlorogenic, and cryptochlorogenic acids; catechin; rutin; and isoquercetin ($p < 0.05$).

3. Results

The statistical descriptors for the levels of total phenolic compounds (TPC), tannins (TTC), flavonoids (TFC), monomeric anthocyanins (TAC), and vitamin C in honeysuckle berries are presented in Table 2. As can be seen, the highest variability was recorded in the case of anthocyanins (CV 34.66%). The variabilities of TPC, TTC, and TFC were in the range of 25–30%; and the lowest variability was that of vitamin C (CV 12.04%). It can be observed that the average level of phenolic compounds quantified in honeysuckle berries (923.98 mg GAE 100 g^{-1}) varied widely, from 516.52 ('Cera' cv., 2020) to 1422.19 mg GAE 100 g^{-1} ('Loni' cv., 2022); and tannins, which represented approximately 58% of the total phenolic compounds, ranged between 354.43 ('Kami' cv., 2020) and 797.00 mg GAE 100 g^{-1} ('Cera' cv., 2022). The average was 535.08 mg GAE 100 g^{-1}.

Table 2. Descriptive statistics for honeyberry total phenolic (TPC), tannin (TTC), flavonoid (TFC), anthocyanin (TAC), and vitamin C *.

	TPC (mg GAE 100 g^{-1})	TTC (mg GAE 100 g^{-1})	TFC (mg CE 100 g^{-1})	TAC (mg C3G 100 g^{-1})	Vitamin C (mg 100 g^{-1})
Average	923.98	535.08	525.58	472.39	63.63
Standard deviation	252.74	139.80	151.33	163.72	7.66
CV (%)	27.35	26.13	28.79	34.66	12.04
Range	905.67	442.57	558.51	563.95	30.66
Minimum	516.52	354.43	332.95	289.89	47.66
Maximum	1422.19	797.00	891.46	853.84	78.32

* Means of three replicates per cultivar per year are presented. CV = variation coefficient.

Flavonoids (56.9% of the group of phenolic compounds) had an average concentration of 525.58 mg CE 100 g^{-1} and reached their lower limit (332.95 mg CE 100 g^{-1}) in 'Kami' (2020), and the higher one (891.46 mg CE 100 g^{-1}) in 'Loni' (2022). In honeysuckle berries, anthocyanins represent the majority group of flavonoids and had an average level of 472.39 mg C3G 100 g^{-1}. Under these conditions, the lowest concentration of anthocyanins (289.89 mg C3G 100 g^{-1}) was recorded in the 'Kami' cv. (2020), and the maximum one (853.84 mg C3G 100 g^{-1}) was reached in the 'Loni' cv. (2022). Vitamin C averaged 63.63 mg 100 g^{-1} and varied between 47.66 ('Kami' cv., 2022) and 78.32 mg 100 g^{-1} ('Loni' cv., 2021). The relationships between the four classes of compounds with antioxidant activity and vitamin C dosed in the study in the fruits of the three cultivars of *L. caerulea* are summarized in Table 3 and indicate that the berries of the lonicera species having high total contents of phenolic compounds also showed high contents of tannins (r = 0.804 ***), flavonoids (r = 0.906 ***), anthocyanins (r = 0.889 ***), and vitamin C (r = 0.596 ***). The highest intense correlation was established between TFC and TAC (r = 0.991 ***), and the least-intense correlations were recorded for vitamin C (r had values between 0.578 ** and 0.623 **).

As shown in Table 4, except for TPC, the cultivar, climatic conditions of the experimental year, and their interaction had significant contributions to the variations in the fruit quality parameters (overall, the effect size—partial eta squared—ranged between 52.9 and 93.1%). TPC content of honeysuckle fruits varied significantly among cultivars (Table 4), from 790.20 mg GAE 100 g^{-1} ('Kami' cv.) to 1149.72 mg GAE 100 g^{-1} ('Loni' cv.), and the fluctuations recorded from one year to another (731.50 mg GAE 100 g^{-1} in 2020 and 1156.94 mg GAE 100 g^{-1} in 2022) had less significance ($p = 0.077$). There was a tendency for the three cultivars to accumulate higher contents of phenolic compounds between 2020 and 2022; therefore, the difference between 'Kami' and 'Cera' was higher in 2021 compared to 2020. Additionally, the most important increase in TPC during the study, 528.56 mg GAE 100 g^{-1}, was the one observed for the 'Cera' cv.

Table 3. Correlation matrix for honeyberry total phenolic (TPC), tannin (TTC), flavonoid (TFC), anthocyanin (TAC), and vitamin C.

		TTC	TFC	TAC	Vitamin C
TPC	Pearson Correlation	0.804 ***	0.906 ***	0.889 ***	0.596 **
	Sig. (*p*)	<0.001	<0.001	<0.001	0.001
TTC	Pearson Correlation	1	0.896 ***	0.888 ***	0.578 **
	Sig. (*p*)		<0.001	<0.001	0.002
TFC	Pearson Correlation		1	0.991 ***	0.595 **
	Sig. (*p*)			<0.001	0.001
TAC	Pearson Correlation			1	0.623 **
	Sig. (*p*)				0.001

*** The correlation is significant at the 0.001 level (two tailed). ** The correlation is significant at the 0.01 level (two tailed). Correlation is significant at the 0.05 level (two tailed).

Table 4. Cultivar and year effects on honeyberry total phenolic (TPC), tannin (TTC), flavonoid (TFC), monomeric anthocyanin (TAC), and vitamin C contents *.

Cultivar/Year			TPC (mg GAE 100 g^{-1})	TTC (mg GAE 100 g^{-1})	TFC (mg CE 100 g^{-1})	TAC (mg C3G 100 g^{-1})	Vitamin C (mg 100 g^{-1})
	'Cera'		832.03 b **	584.03 a	534.85 b	482.29 b	61.59 b
	'Kami'		790.20 b	412.18 b	404.98 c	343.83 c	58.94 c
	'Loni'		1149.72 a	609.04 a	636.91 a	591.06 a	70.35 a
Cultivar		p	<0.001	<0.001	<0.001	<0.001	<0.001
		PES	0.894	0.833	0.908	0.918	0.887
	2020		731.50 c	428.18 c	416.60 c	358.55 c	60.07 c
	2021		883.51 b	531.22 b	494.42 b	435.94 b	67.27 a
	2022		1156.94 a	645.84 a	665.72 a	622.68 a	63.54 b
Year		p	<0.001	<0.001	<0.001	<0.001	<0.001
		PES	0.910	0.837	0.923	0.931	0.740
2020	'Cera'		580.54 a	439.82 ab	413.87 b	357.49 b	55.34 b
	'Kami'		673.55 b	365.08 b	355.00 b	298.87 c	63.15 a
	'Loni'		940.41 b	479.64 a	480.92 a	419.29 a	61.72 a
	Cultivar	p	0.002	0.048	0.009	0.004	<0.001
2021	'Cera'		806.46 b	550.29 a	508.19 b	426.20 b	62.77 b
	'Kami'		708.14 c	394.86 b	372.48 c	327.72 c	61.89 b
	'Loni'		1135.94 a	648.52 a	602.58 a	553.90 a	77.15 a
	Cultivar	p	<0.001	0.004	<0.001	<0.001	<0.001
2022	'Cera'		1109.10 b	761.99 a	682.47 b	663.17 b	66.65 a
	'Kami'		988.90 b	476.60 b	487.47 c	404.89 c	51.79 b
	'Loni'		1372.82 a	698.94 a	827.22 a	799.98 a	72.19 a
	Cultivar	p	0.003	<0.001	<0.001	<0.001	0.001
Cultivar × Year		p	0.077	0.007	0.002	<0.001	<0.001
		PES	0.360	0.529	0.590	0.723	0.874

* Means of three replicates are presented; ** different letters on the columns indicate that the differences between means are significant at the 0.05 level, according to Duncan's multiple range test; *p* values are presented according to the two-way ANOVA analysis of variance (significant at 0.05 level); PES = partial eta squared. *p* values calculated for the cultivar effect's significance in 2020, 2021, and 2022 are presented according to the one-way ANOVA analysis of variance (significant at the 0.05 level).

On average, 'Cera' and 'Loni' cvs. presented similar levels of tannins (TTC) (Table 4), 584.03 and 609.04 mg GAE 100 g^{-1}, respectively. 'Kami' lagged behind (412.18 mg GAE 100 g^{-1}). In general, tannins showed an increasing trend from 2020 (428.18 mg GAE 100 g^{-1}) to 2022 (645.84 mg GAE 100 g^{-1}), and in this condition, the most important increase was recorded for 'Cera'—from 439.82 mg GAE 100 g^{-1}, in the first year, to 761.99 mg GAE

100 g^{-1}, in the last year. The most conservative was 'Kami', with a difference of only 111.52 mg GAE 100 g^{-1}.

The evolution of flavonoids (TFC) (Table 4) was similar to those of polyphenols and tannins: an increase from 2020 (416.60 mg CE 100 g^{-1}) to 2022 (665.72 mg CE 100 g^{-1}), although in this case, the differences between cultivars were higher. Thus, the 'Loni' cv. stood out for its high content of flavonoids (636.91 mg CE 100 g^{-1}) and was followed by the 'Cera' cv., with an average of 534.85 mg CE 100 g^{-1}. The most important increase in TFC between the study years was recorded for 'Loni' cv., from 480.92 to 827.22 mg CE 100 g^{-1}, and the smallest difference was determined for 'Kami'.

In honeysuckle berries, total monomeric anthocyanins (TAC) represented a percentage of 51.1% of TPC and followed an increasing trend between the first and the last experimental year, from 358.55 to 622.68 mg C3G 100 g^{-1} (Table 4). 'Loni' had the highest TAC content, 591.06 mg C3G 100 g^{-1}, followed by 'Cera' cv., which had 482.29 mg C3G 100 g^{-1}. Compared to 'Kami', with a difference of only 106.02 mg C3G 100 g^{-1} between experimental years, the highest increase of 380.69 mg C3G 100 g^{-1} was recorded for 'Loni'. The highest concentration of vitamin C was quantified in 'Loni', 70.35 mg 100 g^{-1}, followed by 'Cera', with 61.59 mg 100 g^{-1}. Regarding the effect of the study year, vitamin C content increased by approximately 12% from 2020 to 2021, followed by a 5.5% reduction over the 2021–2022 period. This dynamic explains the maximum level of vitamin C reached in the 'Loni' cultivar, in 2021, 77.15 mg 100 g^{-1}. Table 5 shows the statistical indicators of the values determined in the honeysuckle cultivars 'Cera', 'Kami', and 'Loni' for lycopene, β-carotene, some phenolic acids (chlorogenic, neochlorogenic, and cryptochlorogenic acids), and flavonoids (catechin, rutin, isoquercetin). It can be seen that, among the studied carotenoids, the level of lycopene was around 0.47 mg/100 g (0.27–0.76 mg 100 g^{-1}), whereas for β-carotene a higher average concentration was determined, 1.27 mg 100 g^{-1} (0.83–1.73 mg 100 g^{-1}).

Table 5. Descriptive statistics of honeyberry lycopene, β-carotene, chlorogenic acid (CA), neochlorogenic acid (NCA), cryptochlorogenic acid (CCA), catechin (C), rutin (R), and isoquercetin (IQ) contents *.

	Lycopene (mg 100 g^{-1})	β-Carotene (mg 100 g^{-1})	CA (mg 100 g^{-1})	NCA (mg 100 g^{-1})	CCA (mg 100 g^{-1})	C (mg 100 g^{-1})	R (mg 100 g^{-1})	IQ (mg 100 g^{-1})
Average	0.47	1.27	72.80	7.77	11.25	164.32	21.59	2.54
SD	0.20	0.34	13.17	2.58	5.73	101.79	6.18	0.38
CV%	42.76	27.03	18.09	33.17	50.94	61.95	28.61	15.14
Range	0.49	0.90	37.47	6.29	13.24	241.20	16.12	1.13
Minimum	0.27	0.83	53.40	4.17	7.06	68.69	14.40	1.95
Maximum	0.76	1.73	90.87	10.46	20.30	309.89	30.52	3.08

* Means of three replicates are presented; SD = standard deviation; CV = variation coefficient.

The most abundant of the phenolic acids was chlorogenic acid, at 72.80 mg 100 g^{-1}, and its concentration ranged from 53.4 ('Cera' cv.) to 90.87 mg 100 g^{-1} ('Loni' cv.). It was followed by cryptochlorogenic acid, at 11.25 mg 100 g^{-1} (7.06–20.30 mg 100 g^{-1}), and neochlorogenic acid, at 7.77 mg 100 g^{-1} (4.17–10.46 mg 100 g^{-1}). Among the flavonoids, catechin predominated with an average of 164.32 mg 100 g^{-1} and great variability, from 68.69 ('Kami' cv.) to 309.89 mg 100 g^{-1} ('Loni' cv.). Rutin had lower concentrations (21.59 mg 100 g^{-1}) and varied widely from 14.40 ('Cera' cv.) to 30.52 mg 100 g^{-1} ('Loni' cv.). Another important compound, isoquercetin, reached an average of 2.54 mg 100 g^{-1} and varied from 1.95 ('Cera' cv.) to 3.08 mg 100 g^{-1} ('Kami' cv.). Except for chlorogenic acid (CV 18.09%) and isoquercetin (CV 15.14%), all other antioxidant compounds presented in Table 5 showed high and very high variabilities (27.03–61.95%).

As shown in Table 6, the correlation between lycopene and β-carotene was negative and insignificant, and the correlation established by β-carotene with catechin was negative and distinctly significant (r = −0.896 **). In the case of lycopene, significant and positive correlations were established with chlorogenic acid (r = 0.876 **), neochlorogenic acid

(r = 0.826 **), and catechin (r = 0.902 **); and the strongest positive correlation was with rutin (r = 0.967 ***). Except for cryptochlorogenic acid, which showed negative correlations with the other representatives of its group and with flavonoids, positive correlations with different degrees of significance were established between chlorogenic and neochlorogenic acids and the three flavonoids, of which, the strongest was between chlorogenic acid and rutin (r = 0.963 ***). Additionally, fruits with high antioxidant activity had high contents of neochlorogenic acid (r = 0.944 ***), chlorogenic acid (r = 0.827 **), isoquercetin (r = 0.828 **), and rutin (r = 0.727 *), and a low content of cryptochlorogenic acid (r = −0.989 ***).

Table 6. Correlation matrix of honeyberry lycopene, β-carotene, chlorogenic (CA), neochlorogenic acids (NCA), cryptochlorogenic (CCA), catechin (C), rutin (R), and isoquercetin (IQ) contents.

		β-Carotene	CA	NCA	CCA	C	R	IQ
Lycopene	Pearson Correlation	−0.638	0.876 **	0.826 **	−0.677 *	0.902 **	0.967 ***	0.631
	Sig. (*p*)	0.064	0.002	0.006	0.045	0.001	*<0.001*	0.068
β-carotene	Pearson Correlation	1	−0.353	−0.153	−0.104	−0.896 **	−0.559	−0.010
	Sig. (*p*)		0.351	0.694	0.790	0.001	0.118	0.980
CA	Pearson Correlation		1	0.963 ***	−0.794 *	0.709 *	0.963 ***	0.876 **
	Sig. (*p*)			*<0.001*	0.011	0.032	*<0.001*	0.002
NCA	Pearson Correlation			1	−0.927 ***	0.562	0.901 **	0.898 **
	Sig. (*p*)				*<0.001*	0.115	0.001	0.001
CCA	Pearson Correlation				1	−0.323	−0.720 *	−0.775 *
	Sig. (*p*)					0.396	0.029	0.014
C	Pearson Correlation					1	0.861 **	0.382
	Sig. (*p*)						0.003	0.310
R	Pearson Correlation						1	0.754 *
	Sig. (*p*)							0.019

*** The correlation is significant at the 0.001 level (two tailed). ** The correlation is significant at the 0.01 level (two tailed). * Correlation is significant at the 0.05 level (two tailed).

Regarding the levels of carotenoids of the three honeysuckle cultivars, it could be observed that the ratio between β-carotene and lycopene varied from 1.25 ('Loni' cv.) to 4.33 (Kami' cv.) (Table 7). The highest quantities of carotenoids (as a sum of lycopene and β-carotene) were recorded in the case of the 'Kami' cv., where the concentration of β-carotene was maximal (1.69 mg 100 g^{-1}), and that of lycopene, which was medium (0.39 mg 100 g^{-1}). 'Loni' presented the highest concentration of lycopene (0.73 mg 100 g^{-1}) but a minimum amount of β-carotene (0.91 mg 100 g^{-1}). Furthermore, the highest variability in the concentration of the two carotenoids was observed for lycopene (42.76%, Table 5), almost 1.5 times higher compared to β-carotene.

Table 7. Cultivar effects on honeyberry lycopene, β-carotene, chlorogenic acid (CA), neochlorogenic acid (NCA), cryptochlorogenic acid (CCA), catechin (C), rutin (R), and isoquercetin (IQ) contents *.

Cultivar	Lycopene (mg 100 g^{-1})	β-Carotene (mg 100 g^{-1})	CA (mg 100 g^{-1})	NCA (mg 100 g^{-1})	CCA (mg 100 g^{-1})	C (mg 100 g^{-1})	R (mg 100 g^{-1})	IQ (mg 100 g^{-1})
'Cera'	0.29 c **	1.21 b	58.19 c	4.51 c	18.82 a	121.50 b	15.54 c	2.11 b
'Kami'	0.39 b	1.69 a	73.32 b	8.60 b	7.61 b	74.60 c	19.98 b	2.71 a
'Loni'	0.73 a	0.91 c	86.90 a	10.19 a	7.33 b	296.86 a	29.26 a	2.80 a
p	<0.001	<0.001	0.001	<0.001	<0.001	<0.001	<0.001	0.026

* Means of three replicates are presented; ** different letters on the columns indicate that the differences between means are significant at the 0.05 level, according to Duncan's multiple range test; *p* values are presented according to the one-way ANOVA analysis of variance (significant at 0.05 level).

The results presented in Table 7 show that 'Loni' had the highest concentrations of CA and NCA (86.90 and 10.19 mg 100 g^{-1}, respectively), followed by 'Kami', with 73.32 mg 100 g^{-1} CA and 8.60 mg 100 g^{-1} NCA. In addition, 'Cera' stood out for its superior content of cryptochlorogenic acid, 18.82 mg 100 g^{-1}, more than twice that of the other two cultivars. The flavonoids identified in *Lonicera caerulea* berries were present in the highest concentration in 'Loni', where 100 g of fruit contained 296.86 mg catechin, 29.26 mg

rutin, and 2.80 mg isoquercetin. With similar levels of isoquercetin (2.71 mg 100 g^{-1}) and cryptochlorogenic acid (7.61 mg 100 g^{-1}) as 'Loni' (Table 7), 'Kami' ranked second in terms of rutin content (19.98 mg 100 g^{-1}) but had a minimum amount of catechin (74.60 mg 100 g^{-1}).

4. Discussions

The results obtained in this study regarding TPC are higher than those reported by Senica et al. [23] for *L. edulis* (1612.61 mg GAE 100 g^{-1} DW), and those from the more recent study of Česonienė et al. [24]: 364–784.5 mg GAE 100 g^{-1}. More similar values were found by Chaovanalikit et al. [25], 427–1140 mg GAE 100 g^{-1} fresh fruit, and by Sic Žlabur et al. [26] (6.209 g GAE 100 g^{-1} DW). Moreover, less TPC variation compared to our study was reported by Shevchuk et al. [27]: from 444 to 1000 mg GAE 100 g^{-1}. The authors highlighted the stronger effect of the cultivar compared to the variability due to the year. This is contrary to our results, in which the effect of the cultivar was distinctly significant, and that of the year varied significantly (data also supported by partial eta squared values).

Tannins are found in large quantities in unripe fruits and are responsible for generating the astringent taste [16] resulting from the interaction between salivary proteins and (hydrolyzable) tannins [28]. Regarding TTC, in a previous study, Mladin et al. [29] determined similar levels of tanned substances in honeysuckle berries, 0.192–0.429% GAE, but with higher variation limits. However, the authors reported that the tannin concentrations in the 'Loni' and 'Cera' cultivars were approximately two time lower as those found in the present study (0.254 and 0.301% GAE), which highlights the influences of the year and the climatic conditions.

The total flavonoid content was close to those reported by Rupasinghe et al. [30] and Raudonė et al. [31]. Rupasinghe et al. [30] reported for the berries of three honeysuckle cultivars ('Borealis', 'Indigo Gem', and 'Tundra') flavonoid concentrations of 594.43–699.29 mg quercetin equivalents (QE) 100 g^{-1} [30]. Flavonoid concentrations between 20,000 μg g^{-1} DW and almost 50,000 μg g^{-1} DW for the cultivars 'Amphora', 'Indigo Gem', 'Leningradskij', 'Nimfa', and 'Tundra'—and also very low concentrations, below 10,000 μg g^{-1} DW in three other cultivars—were reported by Raudonė et al. [31]. Šic Žlabur et al. [26] presented similar flavonoid concentrations (2.825 g GAE 100 g^{-1}) in fresh honeysuckle berries. Regarding the total content of monomeric anthocyanins, the study carried out by Auzanneau et al. [32] reported total anthocyanin levels of 8.4 and 41.1 mg C3G g^{-1} DW, a range in which the values found in our study fell, but without going down to the lower limit mentioned by authors. High variations in the anthocyanin content were found by Raudonė et al. [31], in a study performed on eight cultivars of honeysuckle. The anthocyanin contents found in our study are comparable to those found by Raudonė et al. [31], except for three cultivars mentioned by the authors, where low concentrations of phenolic compounds (below 10,000 μg g^{-1} DW) accounted for their low contents of anthocyanins. A lower anthocyanin content (116 and 593 mg 100 g^{-1} FW) in blue honeysuckle than that found in the present study was reported by Chaovanalikit al. [25]. Other studies referred to anthocyanin contents similar to those reported by Raudonė et al. [31], between 14.3 and 65 mg C3G g^{-1} DW [30,33–36], a range of values that also includes the concentrations of the 'Cera', 'Kami', and 'Loni' cultivars. A recent analysis of 61 honeysuckle genotypes conducted by Fan et al. [37] indicated very high variability of the anthocyanin content of between 158.44 and 1751.44 mg 100 g^{-1}. De Silva and Rupasinghe [38] cited authors who reported anthocyanin contents varying between 68 and 649 mg 100 g^{-1} and found TAC varying between 39.2 and 294 mg C3G 100 g^{-1} in four haskap cultivars, depending on harvest dates. The vitamin C content in honeysuckle berries generally has lower values compared with our results, as reported by Jurikova et al. [39].

Comparing some cultivars and selections of honeysuckle with *Morus nigra*, *Prunus tomentosa*, and *Amelanchier* berries in terms of vitamin C (reported as mg 100 g^{-1} DW), Juríková et al. [39] found the following values: 67.66–186.61 for *Lonicera*, 40.46–96.80 for *Morus*, 123.33 for *Prunus*, and 91.47–114.22 for *Amelanchier*. In further research, Juríková

et al. [40] determined variations in the content of vitamin C in honeysuckle selections of between 9.71 and 46.67 mg 100 g^{-1}, an upper limit that approaches the minimum value reported in the present study. Other studies referred to slightly lower vitamin C contents of 14.55–53.58 mg 100 g^{-1} [24], 17–25 mg 100 g^{-1} [30], or 17.7–35.6 mg 100 g^{-1} [27]. Shevchuk et al. [27] discussed the generally stronger variation in vitamin C content between cultivars rather than between study years, an aspect also found in our study. Šic Žlabur et al. [26] reported higher vitamin C concentrations of 5.348 g 100 g^{-1} in the cultivar 'Indigo Treat' grown in Croatia. Values closer to our results were also determined by Auzanneauet al. [32] in *L. caerulea* cultivars grown in Switzerland (1.78–4.21 mg g^{-1} DW).

Regarding the carotenoid content, higher concentrations of β-carotene compared to the lycopene in honeysuckle berries were previously reported by Palíková et al. [41] (0.720 mg 100 g^{-1} β-carotene), along with less than 0.001 mg 100 g^{-1} of lycopene, similarly to our results. Data available in the literature indicate levels of chlorogenic acid of 2.67–4.98 mg g^{-1} DW [32], or higher, 86.62–267.14 mg 100 g^{-1} DW [39]. Other authors mentioned wide variations in the chlorogenic and neochlorogenic acids content, of 280.31–1222.08 μg CA g^{-1} DW and 23.85–82.74 μg NCA g^{-1} DW [31], which included the values reported for 'Cera', 'Kami', and 'Loni' cultivars. Higher concentrations of chlorogenic acid (539.20 mg 100 g^{-1} DW), but lower concentrations of neochlorogenic acid (19.69 mg 100 g^{-1} DW) and cryptochlorogenic acid (0.57 mg 100 g^{-1} DW) were reported by Senica et al. [42]. In a previous study, Senica et al. [23] mentioned chlorogenic and neochlorogenic acids as the dominant phenolic acids in honeysuckle fruits, an aspect also found in our study as well. Orsavová et al. [43] reported concentrations of chlorogenic acid and rutin compared to catechin in honeysuckle berries. However, the content of chlorogenic acid found (2123.1–4770.8 mg kg^{-1} DW) is closer to those reported for 'Cera', 'Kami', and 'Loni' cultivars. The concentrations of neochlorogenic acid (0.4–21.2 mg kg^{-1} DW), catechin (8.5–93.0 mg kg^{-1} DW), and rutin (20.0–147.8 mg kg^{-1} DW) reported by Orsavová et al. [43] were lower compared to the values recorded in our study.

Other studies referred to the effects of abiotic factors and cited studies that indicated that, along with flavonoids, hydroxycinnamic acids have functions in the interaction of plants with environmental factors such as high-intensity light, extreme temperatures, heavy metals, water stress, etc. [8,10,16,44]. In a recent study, Orsavová et al. [43] and Gołba et al. [16] reported significant variations in the profiles of compounds with antioxidant activity found in honeysuckle berries, depending on the cultivar and cultivation area, and also on the time of ripening. In our study, the strongest effects of the cultivar and the year of study were those related to TAC, followed by TFC and TPC, and the effect of the cultivar × year interaction was observed, especially in the variations of vitamin C content, followed by those of TAC. The greatest variabilities in TPC and TTC were recorded for 'Cera'. TFC varied intensively among the years of study for 'Loni', TAC for 'Cera' and 'Loni' cvs., and vitamin C for 'Kami' and 'Loni' cvs.

5. Conclusions

This study indicated that 100 g of fresh honeysuckle berries from 'Cera', 'Kami', and 'Loni' cultivars contained 519–1422 mg GAE of phenolics, 354.43–797.00 mg GAE of tannins, 332.95–891.46 mg CE of flavonoids, 289.89–853.84 mg of C3G monomeric anthocyanins, 47.66–78.32 mg of vitamin C, 0.27–0.76 mg of lycopene, 0.83–1.73 mg of β-carotene, 53.40–90.87 mg of chlorogenic acid, 7.06–20.30 mg of cryptochlorogenic acid, 4.17–10.46 mg of neochlorogenic acid, 68.69–309.89 mg of catechin, 14.40–30.52 mg of rutin, and 1.95–3.08 mg of isoquercetin. The consumption of fresh honeysuckle berries and the development of processed products, as a valuable source of health-promoting compounds, are of considerable interest and have received more and more attention in recent years due to their bioactive properties. The study of the influences of genotype and year on the phenolic content of three Romanian honeysuckle cultivars confirmed the usefulness of *Lonicera caerulea* species as a rich source of bioactive phenolic compounds with the potential to be used in food and pharmaceutical industries. For these reasons, greater efforts need to

be made to provide new information on the characteristics of *Lonicera caerulea* species to promote its consumption and encourage its cultivation in farms.

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