

Article

Impact of Production Methods and Storage Time on the Bioactive Compounds and Antioxidant Activity of Confitures Made from Blue Honeysuckle Berry (*Lonicera caerulea* L.)

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Abstract: The blue honeysuckle berry is a fruit known as a rich source of many bioactive substances with proven health-promoting effects. Due to its sour taste with a noticeable hint of bitterness, fruits of this plant are rarely consumed and the consumer prefers the processed form. The purpose of this study was to evaluate the effect of the cooking method on the biological quality of honeysuckle berry confiture. The selected recipe was used to make confiture in a vacuum evaporator using lowered pressure and in a thermomix vessel under atmospheric pressure. Then, the content of the chosen compounds and antioxidant activity of the two types of confitures were compared. The confitures were analyzed right after production and through 180 days of refrigerated storage. The pH, TA and TSS parameters remained unchanged regardless of the production process and storage time. Ascorbic acid, polyphenol and anthocyanin concentrations were greater in the confiture from vacuum cooking. Also, the same confiture showed a lower rate of degradation of bioactive substances during storage. The antioxidant activity of the two types of confiture was significantly different shortly after production, but equal at the end of 180-day storage. HMF content was four times higher in confitures cooked under atmospheric pressure than under vacuum. The confiture made from the honeysuckle berry was very rich in bioactive compounds, especially polyphenols. Vacuum cooking proved to be the best method for confiture production as a result of lower temperatures used and less aeration of the mass.

Keywords: fruit preserves quality; vacuum cooking; single-phase method; ascorbic acid; anthocyanins; total polyphenols content; HMF



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1. Introduction

A valuable raw material for the production of fruit preserves with increased health-promoting value is blue honeysuckle berry (*Lonicera caerulea* L.). Recently, this fruit has been gaining popularity in the food industry. It is native to Siberia and northern Asia, but is currently cultivated in many parts of the world [1,2]. The largest plantations are located in China and North Korea (2000 ha), Poland (1800 ha), Canada (1000 ha), Russia (400 ha) and Japan (160 ha) [3]. However, Poland was the world's production leader in the year 2020 [4].

The sugar profile of this fruit is dominated by glucose and fructose, but also contains sucrose and sorbitol. The honeysuckle berry includes other valuable elements and compounds such as calcium, magnesium, fiber, iridoids and polyunsaturated fatty acids. In addition, honeysuckle contains anthocyanins and other polyphenols, as well as vitamin C and E, which together are responsible for high antioxidant activity [2,4–6]. A study by Orsavová et al. [2] indicates that, depending on the variety and location of

cultivation, honeysuckle berries contain, in 100 g of dry weight, about 1752–5408 mg of polyphenols, 171–568 mg of anthocyanins and 1628–2855 mg of vitamin C. Among the colorants responsible for the deep, blue color of honeysuckle, the cyanidin-3-O-glucoside dominates, accounting for 79–92% of all anthocyanins [7]. The rest of the anthocyanin compounds include cyanidin-3-O-diglucoside, cyanidin-3-O-rutinoside, pelargonidin-3-O-glucoside and peonidin-3-O-glucoside [8]. Numerous health-promoting compounds present in honeysuckle berry and its products contribute to their anti-ageing, cardioprotective, neuroprotective, anti-inflammatory, antidiabetic or antimicrobial properties [1,2,5–7]. In addition, the berry is known to counteract other civilization diseases. Cyanidin-3-O-glucoside extracted from these fruits has been shown to be a substance with high potential to inhibit pulmonary carcinogenesis [9], and has a significant role in the antiproliferation of breast and liver cancer cells [10]. Honeysuckle berry consumption, thanks to its richness in polyphenols, can prevent obesity by reducing fat absorption, as well as positively modifying the gut microbiota [11].

Blue honeysuckle berry is characterized by a distinctive, astringent taste; hence, the organoleptic acceptance of raw fruits may be limited. Therefore, it is better accepted by the average consumer in processed form. From honeysuckle berries are produced juices, infusion blends, spreads, smoothies, wines, jams, candies, ice cream, yoghurt, jellies, pastries, and dried fruits [6,8]. In Poland, popular honeysuckle berry products include preserves like jams or confitures, which can be a rich source of fruits and bioactive compounds in a varied diet.

Due to the developing awareness of food quality, alternative methods of food production and processing are being explored in order to preserve the best possible organoleptic features and a significant amount of bioactive compounds. It is important to inhibit or counteract the negative thermally induced biochemical changes that lead to the formation of harmful compounds, such as hydroxymethylfurfural (HMF) and overall product quality deterioration, as much as possible. For the production of jams and other preserves, a concentration process under reduced pressure allows simultaneous reduction of temperature and limitation of oxygen effects on bioactive compounds, as well as shortening treatment duration. There have been scientific studies to identify strategies for reducing HMF formation in food using vacuum treatment [12]. Tomruk et al. [13] reported that strawberry jams obtained by vacuum cooking had a better, more intense red color and from two to nine times less HMF than jams after conventional atmospheric cooking. Vacuum-evaporation-made jam from jujube fruit contained 20.61% more polyphenols compared to open-pan-evaporation jam [14]. Bilberry jams cooked using the vacuum method showed higher average antioxidant activity, anthocyanins and vitamin C content, as well as better color than jams obtained using the conventional method [15].

In view of the above, as well as a result of the lack of scientific literature on honeysuckle berry preserve quality, the aim of this study was to evaluate the effect of different production methods on the quality characteristics of honeysuckle low-sugar confiture; in particular, the content of vitamin C, polyphenols, anthocyanins and HMF. The antioxidant capacity and basic physicochemical parameters were also evaluated. In addition, storage tests were also conducted to determine the stability of individual compounds.

2. Materials and Methods

2.1. Plant Material

The research material consisted of blue honeysuckle berry (*Lonicera caerulea* L.) fruits of the variety Wojtek. The fruits were harvested in the year 2021 in late June at a plantation owned by Nutraceut Sp. z o.o., located in Poland in the Kuyavian–Pomeranian Voivodeship. They were harvested 5 days after full coloring, previously evaluated in terms of firmness, organoleptic characteristics and extract by plantation workers. Then, fruits were transported to the laboratory under refrigerated conditions and immediately used for our research.

2.2. Reagents and Solvents

Sodium hydroxide, gallic acid, Folin–Ciocalteu reagent, sodium carbonate, formic acid, oxalic acid and Carrez I and II reagent, were reagent-grade, whereas acetonitrile, methanol and orthophosphoric acid were of HPLC grade. All reagents and solvents were purchased from Merck KGaA (Darmstadt, Germany). Ultra-pure water with 18.2 MΩ cm resistivity was obtained using the Milli-Q[®] system (Merck Millipore, Darmstadt, Germany).

2.3. Production of Blue Honeysuckle Berry Confitures

The research material consisted of low-sugar confitures from blue honeysuckle berries obtained in two variants which differed in the method of production. The I variant was obtained in a thermomix under atmospheric pressure and II variant was obtained by vacuum evaporator, all made using the same recipe (R3), selected in preliminary studies (Table 1). Throughout the entire study, confitures were obtained using the single-phase method. The single-phase method is currently considered the most popular on an industrial scale, due to time savings and high product quality.

Table 1. Recipe ingredients of studied confitures (% content).

Ingredient	R1	R2	R3	R4	R5
blue honeysuckle berry	63.7	63.7	63.7	63.7	63.7
sugar	28.85	28.7	28.55	28.45	28.35
water	7.2	7.2	7.2	7.2	7.2
pectin	0.25	0.25	0.25	0.25	0.25
guar gum	0	0.15	0.30	0.40	0.50

In the case of obtaining confitures in the thermomix (Vorwerk, Wuppertal, Germany), sugar and water were heated for 5 min at 60 °C until the sugar was dissolved. Then, fruit was added and cooked at 100 °C for 30 min. Next, 5 min before the end of cooking, guar gum and low-methylated pectin were added. To obtain confitures in a vacuum evaporator (Rotavapor[®] R-124, BÜCHI Labortechnik AG, Flawil, Switzerland), sugar syrup was prepared, fruit was added and the entire mixture was cooked at 55 °C under reduced pressure of 150 mbar, with guar gum and low-methylated pectin added 5 min before the end of processing. The cooking process was always terminated when the final product obtained an extract of 40%. In both variants, after cooking, the confitures were placed in 40 mL jars, pasteurized for 10 min at 85 °C and then cooled to 20 °C. The confitures were subjected to storage for 180 days at 6 °C.

2.4. Physicochemical Parameters Analysis

The prepared confitures were subjected to basic physicochemical analyses. Active acidity (pH) was determined using a calibrated electric pH meter (HI 221, Hanna Instruments Inc., Smithfield, RI, USA). Titratable acidity (TA) was determined using the automatic titrator TitroLine[®] 5000 (SI Analytics, Mainz, Germany). The results were expressed as g of citric acid per 100 g of confitures. Total soluble solids (TSS) were determined using a refractometer Refracto 30PX (Mettler Toledo, Columbus, OH, USA). The results were expressed in °Brix.

2.5. Total Phenolics, Anthocyanins and Antioxidant Activity Determinations

2.5.1. Preparation of Extracts

To analyze the polyphenol content of the confiture samples, appropriate extraction was performed. First, 1 g of confiture was combined with 5 mL of extraction mixture (80% methanol acidified with 0.1% HCl). Samples were exposed to ultrasound (SW-3H, Sonoswiss AG, Ramsen, Germany) for 3 min, followed by centrifugation for 10 min at 12,000 rpm (MPW-350R, MPW MED. INSTRUMENTS, Warszawa, Poland). Samples were extracted repeatedly until discoloration. Supernatants were gathered into a 50 mL volumet-

ric flask after each centrifugation step. After extraction, the flasks were filled to the volume with the extraction mixture.

2.5.2. Total Polyphenol Content Analysis

Total polyphenol content (TPC) of samples was determined using Folin–Ciocalteu reagent's method [16]. Briefly, 0.2 mL of extract prepared as described in Section 2.5.1 was mixed with 0.4 mL of Folin–Ciocalteu reagent, 4 mL of redistilled water and 2 mL of 15% sodium carbonate. After 60 min of solution incubation in a dark place at the ambient temperature, the absorbance determinations were taken with a UV-1650C spectrophotometer (Schimadzu, Kyoto, Japan) at 765 nm against mixed reagents. A five-point standard curve was prepared based on 59 mM stock solution of gallic acid in 80% methanol ($y = 0.3938 + 0.107x$, $R^2 = 0.9995$). The results were expressed as mg gallic acid equivalents (GAE) per 100 g of confiture sample.

2.5.3. Chromatographic Analysis of Anthocyanins

The anthocyanin content in the prepared variants of confitures was determined using a high-pressure liquid chromatography with diode array detector (Prominence HPLC System, Shimadzu, Japan). The measurement was performed in accordance with already published methodology [17]. The extracts were passed through a 0.45 μm PTFE syringe filter into vials. The separation of anthocyanins was conducted on a C18(2) Luna column (100 \AA , 5 μm , 250 \times 4.6 mm) from Phenomenex (Torrance, CA, USA). The mobile phase was a water/acetonitrile/formic acid mixture in a volumetric ratio of 81:9:10. An isocratic flow at a flow rate of 1 mL/min was used. The chromatograms were recorded at a $\lambda = 520$ nm. The anthocyanin content was expressed as mg of cyanidin-3-O-glucoside per 100 g.

2.5.4. Determination of Antioxidant Activity

Antioxidant activity was determined with the radical scavenging activity (DPPH) method described by Yen and Chen [18] with some modifications. The working DPPH solution was obtained by dilution of stock solution (12 mg of DPPH in 100 mL of methanol) to obtain absorbance of 0.4. To 1 mL of the extract prepared as described in Section 2.5.1, 3 mL of methanol and 1 mL of DPPH working solution were added and mixed. The absorbance of the reagent mixture at $\lambda = 517$ nm was measured accurately after 10 min using UV1650PC spectrophotometer (Schimadzu, Japan).

The antioxidant activity was determined also with cation radical scavenging activity (ABTS) according to the method of Re et al. [19]. Briefly, 40 μL of extract was mixed with 4 mL of ABTS solution. Six minutes after the addition of the ABTS solution, the absorbance of samples was measured using a UV1650PC spectrophotometer (Schimadzu, Japan) in relation to distilled water at $\lambda = 734$ nm.

The results from both determinations were calculated using a calibration curve prepared at different concentrations of Trolox in methanol and expressed as μM Trolox/g of confiture.

2.6. Chromatographic Analysis of Ascorbic Acid

A total of 4 g of confiture sample was placed in a beaker with 2% oxalic acid, and then homogenized on T 25 digital ULTRA-TURRAX[®] (IKA[®] Poland Sp. z o.o., Lozienia, Poland). After homogenization, the samples were transferred to 50 mL volumetric flasks, filled to the volume with 2% oxalic acid, and mixed. The samples were exposed to ultrasound (SW-3H, Sonoswiss AG, Germany) for 5 min and then centrifuged for 10 min at 14,000 rpm (MPW-350R, MPW MED. INSTRUMENTS, Poland). The supernatants were passed through a 0.45 μm PTFE syringe filter into vials.

The ascorbic acid content was determined using a high-pressure liquid chromatography coupled with UV-VIS detector (Prominence HPLC System, Shimadzu, Japan), according to a previously published method [20]. The stationary phase was an Onyx Monolithic C18 column (100 \times 4.6 mm, 5 μm , Phenomenex, Torrance, CA, USA). The mobile phase was a

0.1% m-phosphoric acid in an isocratic flow at a flow rate of 1 mL/min. The chromatograms were recorded at $\lambda = 254$ nm. The results were expressed as mg of ascorbic acid per 100 g.

2.7. Chromatographic Analysis of Hydroxymethylfurfural

To prepare the extracts, 4 g of confiture was placed in a 25 mL volumetric flask, then reagents 1 mL of each Carrez I and II was added, and flask was filled up with redistilled water and mixed. The resulting mixtures were centrifuged for 10 min at 4500 rpm (MPW-350R, MPW MED. INSTRUMENTS, Poland), and supernatants were passed through a 0.45 μm PTFE syringe filter into vials.

Total hydroxymethylfurfural (HMF) content in confiture samples was determined using a high-pressure liquid chromatography with UV-VIS detector (Prominence HPLC System, Shimadzu, Japan), in accordance with the methodology reported by Rada-Mendoza et al. [21] with our own modifications that involved the change of the gradient elution to isocratic flow. Briefly, separation of HMF was conducted on a C18(2) Luna column (100 \AA , 5 μm , 250 \times 4.6 mm) from Phenomenex (Torrance, CA, USA). The mobile phase was a methanol/water mixture in a ratio of 1/9 *v/v*. An isocratic flow was used at a flow rate of 1 mL/min. The chromatograms were recorded at a $\lambda = 285$ nm. The HMF content was expressed as mg per 100 g.

2.8. Statistics

The production of honeysuckle berry confitures was performed in two independent repetitions. All presented determinations were carried out in triplicate and presented as a mean with standard deviation. Statistical analysis was performed in Statistica 13.3 (TIBCO Software Inc., Carlsbad, CA, USA). The effects of the different processing treatment on the measured compounds content were determined using ANOVA analysis of variance. The differences between means were evaluated with the Tukey HSD post hoc test ($\alpha = 95\%$).

3. Results and Discussion

3.1. Selection of Optimal Confiture Recipe

Prior to the main study, the most optimal confiture recipe was selected on the basis of sensory evaluation. The evaluation was carried out by a team of 14 people, properly trained, with a 5-point scale. Six confiture recipes were evaluated, in which the dose of guar gum varied from 0 to 0.5%. Considering the fact that confiture is a common addition to desserts such as pancakes or baked goods, consistency plays a critical role in this product. In the sensory evaluation, the consistency of the confitures obtained with 0.3% guar gum addition (R3) was considered the most optimal, while the confitures obtained without this additive had the worst ratings (Figure 1). The addition of guar gum modified the properties of the resulting confiture gel and had a stabilizing effect on the matrix. The R3 confiture variant also scored the best with regard to color and flavor.

Based on these observations, we decided to choose the R3 recipe variant with 0.3% addition of guar gum to achieve the right consistency for the product and to prevent syneresis. Further research on the influence of production methods on the quality of confiture was carried out using only the R3 recipe.

3.2. Physicochemical Parameters

Analysis of the results showed that there was no statistically significant difference ($p < 0.05$) in the values of the pH, TA and TSS parameters between the two methods of confiture production. Regardless of the method of confiture production (thermomix or evaporation under reduced pressure), the pH value of the product was 3.0 ± 0.2 , the acidity was 1.0 ± 0.1 g of citric acid/100 mL and the TSS was $40 \pm 1^\circ$ Brix. Moreover, during the entire storage period, these parameters remained consistent. The lack of changes in physicochemical parameters was mainly due to similar production times using two different methods. In both cases, there was no hydrolysis or degradation of sugars, which are mostly responsible for the formation of TSS. Furthermore, the low temperature at which

the confitures were stored in airtight vessels allowed the content of organic acids and sugars to remain at similar levels.

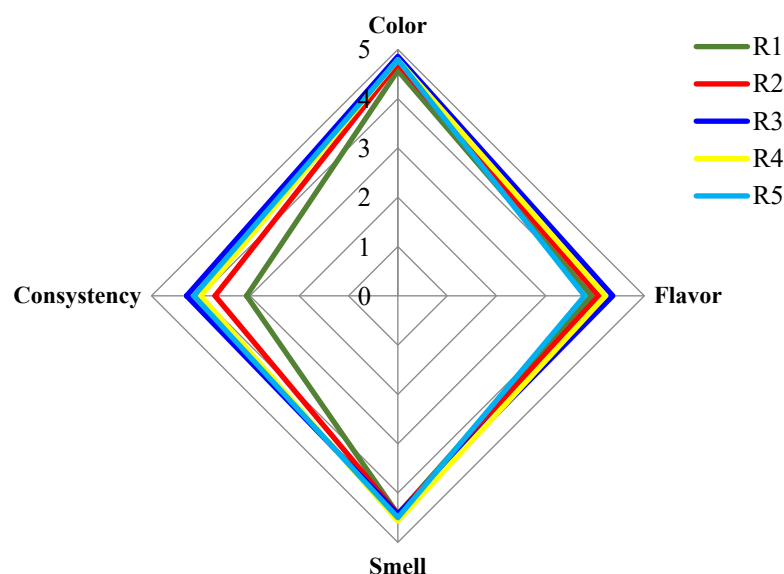


Figure 1. The scores of the sensory evaluation of different confitures recipes. R1—recipe without guar gum; R2, R3, R4, R5—recipes with 0.15, 0.30, 0.40, 0.50% of guar gum content.

It is recommended that the pH of fruit preserves should be in the range of 2.8 to 3.2 to ensure effective gelation of pectin [14]. Grobelna et al. [22] observed lower pH in honeysuckle berry puree in the range of 2.6–2.9, depending on the variety and heat treatment time. Studies on strawberry jams obtained at atmospheric pressure and reduced pressure suggested no significant effect of pressure on pH and TA [13]. Other researchers obtained significantly higher TSS and pH values in apple pekmez using open-pan evaporation method, while no change was observed in TA [23]. Depending on the cooking time used, there may be significant differences between jams concentrated by the two methods. The extract of jujube jam that was vacuum-heated for 20 min differed significantly from jam that was subjected to 15 min of cooking at atmospheric pressure [14]. Similar observations to those of our work were indicated by Devseren et al. [24]—the production of tomato paste by vacuum and with atmospheric evaporation did not result in differences in TSS of the final product.

3.3. Ascorbic Acid Content

The confitures produced in an open vessel as well as under reduced pressure were characterized by similar ascorbic acid content (Table 2). Korus et al. [15] obtained a considerably greater difference in vitamin C content in their jams as a result of the different production process. They indicated that bilberry jams concentrated under vacuum contained from 8.6 to even 18.3% more vitamin C than those concentrated conventionally, which is likely related to the different temperature levels. Depending on the variety of blue honeysuckle berry used, the puree made from them contained less ascorbic acid (10.7 to 19.5 mg per 100 g) [22] than our confitures.

The honeysuckle berry fruits contain more ascorbic acid than processed products due to the degradation of this compound during heat treatment and presence of oxygen. Regarding the berry origin or variety, the literature indicates different content of ascorbic acid in fresh honeysuckle fruits: 1628–2855 mg in 100 g dry weight (Czech Republic) [2], 178–421 mg/100 g fresh weight (Canada) [25], 250 mg/100 g fresh weight (Portugal) [5] or 90–300 mg/100 g fresh weight (Poland) [4].

Table 2. Impact of processing methods and storage time on the bioactive compounds content in blue honeysuckle berry confitures.

Confitures	Days of Storage	Ascorbic Acid (mg/100 g)	TPC (mg GAE/100 g)	HMF (mg/100 g)
From Thermomix (Normal Pressure)	0	23.7 ± 0.4 ^{aA}	431 ± 6 ^{aB}	0.55 ± 0.01 ^{cA}
	30	18.5 ± 0.2 ^{bB}	350 ± 3 ^{bB}	0.59 ± 0.01 ^{abA}
	120	11.2 ± 0.3 ^{cB}	335 ± 8 ^{bcA}	0.63 ± 0.04 ^{abA}
	180	6.1 ± 0.2 ^{dB}	322 ± 9 ^{cA}	0.67 ± 0.05 ^{aA}
From Vacuum Evaporator (Lowered Pressure)	0	23.5 ± 0.7 ^{aA}	501 ± 7 ^{aA}	0.14 ± 0.01 ^{aB}
	30	20.8 ± 0.4 ^{bA}	378 ± 4 ^{bA}	0.15 ± 0.01 ^{aB}
	120	17.6 ± 0.3 ^{cA}	351 ± 7 ^{cA}	0.16 ± 0.01 ^{aB}
	180	12.4 ± 0.2 ^{dA}	332 ± 3 ^{dA}	0.17 ± 0.01 ^{aB}

TPC—total phenolic content; HMF—hydroxymethylfurfural; values in the same column marked with different small letters are significantly different ($p < 0.05$) and concern changes during storage for specific production method; values in the same column marked with different capital letters are significantly different ($p < 0.05$) and concern data comparison between processing methods for specific day of storage.

In both types of confitures, ascorbic acid has gradually degraded. However, in confiture cooked under reduced pressure, the degradation was significantly lower, and finally after 180 days of cold storage it contained about two times more ascorbic acid than other confiture types. A similar effect of the vacuum cooking method was found by other researchers in bilberry jams [15]. This was probably due to the lower oxygen concentration in the confiture mass, which causes oxidation of ascorbic acid [20]. In addition, the low storage temperature allows the preservation of more ascorbic acid [26].

3.4. Total Polyphenol Content

The content of polyphenols in food varies and is influenced by numerous internal and external parameters, including the temperature used, light exposure, oxygen, enzymes, proteins, metal ions or bonding with other food components [27,28].

The total polyphenol content (TPC) in the studied variants of confitures significantly depended on the method of production (Table 2). Immediately after production, the jams obtained under lowered pressure contained 16% higher TPC than those obtained under atmospheric pressure in the thermomix. Also in a study on bilberry jams with herbs, the use of vacuum during production was shown to preserve 19–23% more polyphenols compared to cooking in an open pan [15].

The 180-day cold storage of both types of honeysuckle berry confitures resulted in a significant reduction in TPC. However, the rate of polyphenol degradation varied. A higher rate of degradation changes was noted in confiture from the evaporator where 77% of the initial amount of polyphenols remained. After the same storage time, 75% of the initial amount of polyphenolic compounds remained in the confiture from the thermomix. Nevertheless, at the end of storage, both types of confitures showed similar TPC values. The reduction in polyphenol levels during storage depends on the type of product, time and storage conditions. In another study, after 20 weeks of storage of black carrot jams, a 26.4–48.0% and 21.0–42.5% decrease in TPC was observed in samples stored at 25 °C and 4 °C, respectively [29]. Other researchers noted 17.6 and 36.8% TPC losses in strawberry jams after 28 days of storage at 4° and 15 °C, respectively [26]. During confiture or jam cooking, the cellular structure is disrupted and the raw material becomes susceptible to non-enzymatic oxidation, which can be one of the main causes of loss of phenolic compound content. On the other hand, the heat treatment used during the processing of jams or confitures can cause structural changes that ultimately translate into greater bioavailability of polyphenols [29].

3.5. Hydroxymethylfurfural Content

The confitures prepared in the vacuum evaporator compared to the ones made at normal pressure in the thermomix had significantly lower HMF content directly after production, as well as after 30, 120 and 180 days of cold storage (Table 2). This is undoubtedly an advantage of the vacuum method and confirms the relevance of the research conducted in the context of obtaining a better-quality product. The crucial factor for such significant discrepancies in the content of this compound was the level of temperature applied during a similar heating time. Right after production, the HMF content in our confitures from the vacuum evaporator and thermomix was 0.14 and 0.55 mg/100 g, respectively. Tomruk et al. [13] noted, in strawberry jams cooked in vacuum and atmospheric conditions, the HMF quantity ranges of 1.44–2.14 mg and 4.94–12.90 mg in 100 g, respectively. In another study, nine times more HMF was found in jujube jam prepared using an open-pan evaporation method than by vacuum evaporation [14].

During the 180 days of storage, HMF content increased slightly to 0.17 and 0.67 mg/100 g in the evaporator and thermomix confitures, respectively. In addition, similar dynamics of changes in HFM content were observed between the different storage periods: 0–30 days, 30–120 days and 120–180 days. Other researchers also report increased HMF levels in fruit preserves stored under various conditions. In some cases, the increase in HMF in the product was significantly higher than that recorded by us. A 120-day cold storage of bitter orange jam and orange jam resulted in a 32.9% and 112.7% increase in HMF content [30], while our blue honeysuckle berry confitures showed only 14.5% (thermomix) and 14.3% (evaporator) increases over the same period of time. Djaoudene and Louaileche [31] observed an increased content of HMF during 30-day storage of orange jam. Its levels increased by 144% and 169% during storage at 25 °C and 35 °C, which is about a 20-fold change compared to our own study. This indicates that not only the temperature of the processing, but also the temperature of the storage can significantly affect this HMF quantity in fruit preserves.

3.6. Anthocyanins Content

Anthocyanins are among the compounds that determine the color of a product, as well as their health-promoting properties. The anthocyanin content of the final product depends on the type of raw material used and the method of processing and preservation. In our study, a significant effect of the production method on the content of the anthocyanins was shown (Table 3). Directly after production, the confitures obtained in the vacuum evaporator contained about 37% more anthocyanins than those obtained in the thermomix. Depending on the variety, the total concentration of anthocyanins in blue honeysuckle berry fruits ranges from 86 to 655 mg/100 g fresh weight [6,32,33]. Anthocyanins constitute on average 94% of the total content of polyphenolic compounds present in these fruits [32].

Four anthocyanin monomers were identified in the investigated confitures. The predominant anthocyanin was cyanidin-3-glucoside, accounting for 90% of all anthocyanins. Subsequently, cyanidin-3-rutinoside accounted for 6% of all anthocyanins, and cyanidin-3,5-diglucoside for nearly 3%. Also, peonidin-3-glucoside was identified, which accounted for less than 1%. The shares of individual anthocyanins were within the limits normally reported in the literature [7,34].

A reduction in anthocyanin content was observed in both types of confitures during refrigerated storage (Table 3). After 180 days, 74% of the initial anthocyanin content remained in the confitures cooked in the thermomix, and 60% in the confitures concentrated in the vacuum evaporator. However, the final anthocyanin content of confitures from the vacuum evaporator was 10% higher than that of the corresponding confitures from the thermomix. The rate of transformations of anthocyanins depends on a number of factors such as initial concentration or chemical structure. During storage, anthocyanins primarily undergo polymerization and condensation processes with other polyphenols and phenolic acids [35].

Table 3. Impact of processing methods and storage time on the anthocyanin content (mg of cyanidin-3-O-glucoside/100 g) in blue honeysuckle berry confitures.

Anthocyanin	From Thermomix (Normal Pressure)				From Vacuum Evaporator (Lowered Pressure)			
	0 day	30 day	120 day	180 day	0 day	30 day	120 day	180 day
Cy-3.5-diglu	6.4 ± 0.4	5.4 ± 0.5	4.4 ± 0.2	4.2 ± 0.1	8.4 ± 0.3	5.9 ± 0.2	4.7 ± 0.2	4.3 ± 0.2
Cy-3-glu	224.4 ± 7.5	184.4 ± 5.9	176.1 ± 4.4	167.9 ± 3.4	309.3 ± 19.2	223.3 ± 5.2	203.4 ± 9.5	186.1 ± 5.5
Cy-3-rut	14.8 ± 0.3	11.6 ± 1.4	11.2 ± 0.6	10.4 ± 0.7	19.9 ± 0.5	13.3 ± 0.5	12.0 ± 0.1	10.8 ± 0.7
Pn-3-glu	2.2 ± 0.3	1.8 ± 0.1	1.6 ± 0.1	1.6 ± 0.1	3.0 ± 0.1	2.3 ± 0.2	2.0 ± 0.1	1.8 ± 0.9
Total	248.5 ± 7.9 ^{aB}	203.8 ± 6.3 ^{bB}	193.7 ± 4.0 ^{bB}	184.5 ± 2.7 ^{cB}	341.5 ± 19.3 ^{aA}	245.3 ± 5.8 ^{bA}	222.7 ± 9.3 ^{bA}	203.4 ± 4.6 ^{cA}

Cy-3.5-diglu: cyanidin-3.5-O-diglucoside; Cy-3-glu: cyanidin-3-O-glucoside; Cy-3-rut: cyanidin-3-O-rutinoside; Pn-3-glu: peonidin-3-O-glucoside; values in the same row marked with different small letters are significantly different ($p < 0.05$) and concern changes during storage for a specific production method; values in the same row marked with different capital letters are significantly different ($p < 0.05$) and concern data comparison between processing methods for specific day of storage.

3.7. Antioxidant Activity

The presence of polyphenols in high amounts, including anthocyanins, resulted in the high antioxidant activity of the obtained confitures. Antioxidant capacity against the DPPH radical determined directly after production was 42% higher for confitures cooked in a vacuum evaporator (Figure 2). However, in the same confitures, the antioxidant activity decreased significantly during storage, and the final values for both types of confitures equalized. This trend is consistent with the changes observed for the polyphenols and anthocyanins discussed earlier. According to studies performed on marmalades from various fruit species, there is a high correlation between the content of total polyphenols and antioxidant activity in such kinds of products [36].

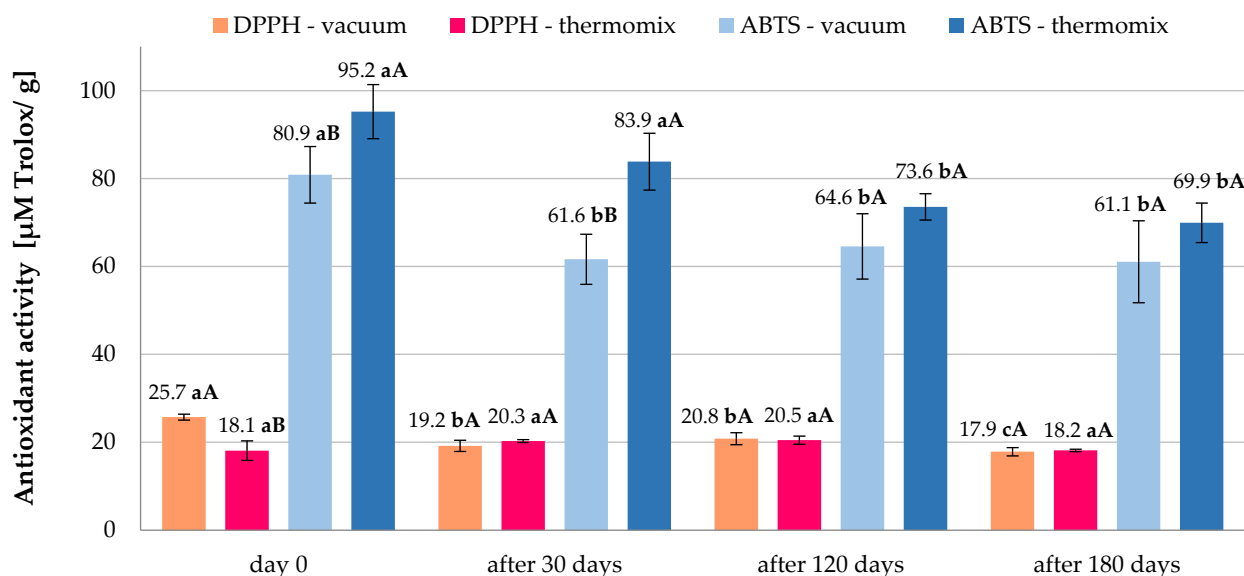


Figure 2. Results of antioxidant activity analyses using DPPH and ABTS methods in studied confitures. Values at the bars of the same color marked with different small letters are significantly different ($p < 0.05$) and concern changes during storage for a specific production method; values at the bars of similar color hue marked with different capital letters are significantly different ($p < 0.05$) and concern data comparison between processing methods for a specific day of storage.

Due to the various reaction mechanisms against different types of radicals, and sensitivity to different-molecular-weight phenolics, the antioxidant activity of food is often measured by several methods simultaneously [37]. Antioxidant activity against the ABTS radical was determined, and after production the confiture from the thermomix showed a higher level (Figure 2). After 180 days of cold storage, the antioxidant activity decreased, but statistically the final values for both types of confitures equalized. The antioxidant

activity of jams made from different fruit species can vary greatly; for example, blueberry 6.1, raspberry 10.1, blackberry 18.3, cranberry 20.2 or blackcurrant 36.6 μM Trolox/g of jam [38]. The mentioned jams contained 50% of fruits in the recipe, while our confitures had 64% fruit content, but even taking this difference into account, it can be concluded that blue honeysuckle berry confiture was distinguished by very high antioxidant activity (80.9–95.2 μM Trolox/g).

4. Conclusions

The use of atmospheric or reduced pressure methods for confiture production has no significant effect on physicochemical values like pH, TA, TSS, and ascorbic acid content. As a result of this research, it has been proven that blue honeysuckle berry confitures are a good source of bioactive components such as polyphenols. Confitures prepared in a vacuum evaporator were characterized by a higher content of total polyphenols and anthocyanins after production as well as during the initial storage period, where the dynamics of degradation changes were the highest. After six months of cold storage, regardless of the method of manufacture, the content of polyphenols and anthocyanins in confitures was at similar level. Furthermore, blue honeysuckle berry confitures, especially those from the vacuum evaporator, were characterized by a low content of HMF. When we put together all the observations, it can be concluded that the confiture produced under reduced pressure in a vacuum evaporator is more favorable in terms of overall quality.

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References

1. Grobelna, A.; Kalisz, S.; Kieliszek, M.; Giurgiulescu, L. Blue honeysuckle berry (*Lonicera caerulea* L.), as raw material, is particularly predisposed to the production of functional foods. *Carpathian J. Food Sci. Technol.* **2020**, *12*, 144–155. [[CrossRef](#)]
2. Orsavová, J.; Sytařová, I.; Mlček, J.; Mišurcová, L. Phenolic compounds, vitamins C and E and antioxidant activity of edible honeysuckle berries (*Lonicera caerulea* L. var. kamtschatica Pojark) in relation to their origin. *Antioxidants* **2022**, *11*, 433. [[CrossRef](#)]
3. Cassels, L.J. Experiences and conclusions from the last seven years of North American haskap cultivation: Varieties, fertilization and market trends. In Proceedings of the Haskap Conference 2017, Ozarów Mazowiecki, Poland, 9 November 2017.
4. Gawroński, J.; Zebrowska, J.; Pabich, M.; Jackowska, I.; Kowalczyk, K.; Dyduch-Siemirńska, M. Phytochemical characterization of blue honeysuckle in relation to the genotypic diversity of *Lonicera* sp. *Appl. Sci.* **2020**, *10*, 6545. [[CrossRef](#)]
5. Molina, A.K.; Vega, E.N.; Pereira, C.; Dias, M.I.; Heleno, S.A.; Rodrigues, P.; Fernandes, I.P.; Barreiro, M.F.; Kostić, M.; Soković, M.; et al. Promising antioxidant and antimicrobial food colourants from *Lonicera caerulea* L. var. Kamtschatica. *Antioxidants* **2019**, *8*, 394. [[CrossRef](#)]
6. Gołba, M.; Sokół-Łętowska, A.; Kucharska, A.Z. Health properties and composition of honeysuckle berry *Lonicera caerulea* L. An update on recent studies. *Molecules* **2020**, *25*, 749. [[CrossRef](#)]
7. Rupasinghe, H.P.V.; Arumuggam, N.; Amararathna, M.; De Silva, A.B.K.H. The potential health benefits of haskap (*Lonicera caerulea* L.): Role of cyanidin-3-O-glucoside. *J. Funct. Foods* **2018**, *44*, 24–39. [[CrossRef](#)]
8. Khattab, R.; Ghanem, A.; Brooks, M.S.-L. Stability of Haskap berry (*Lonicera caerulea* L.) Anthocyanins at different storage and processing conditions. *J. Food Res.* **2016**, *5*, 67. [[CrossRef](#)]

9. Amararathna, M.; Hoskin, D.W.; Rupasinghe, H.P.V. Cyanidin-3-O-glucoside-rich Haskap berry administration suppresses carcinogen-induced lung tumorigenesis in A/JCr Mice. *Molecules* **2020**, *25*, 3823. [[CrossRef](#)]
10. Pace, E.; Jiang, Y.; Clemens, A.; Crossman, T.; Rupasinghe, H.P.V. Impact of thermal degradation of cyanidin-3-O-glucoside of haskap berry on cytotoxicity of hepatocellular carcinoma HepG2 and breast cancer MDA-MB-231 cells. *Antioxidants* **2018**, *7*, 24. [[CrossRef](#)]
11. Kim, J.Y.; Lee, Y.S.; Park, E.J.; Lee, H.J. Honeysuckle berry (*Lonicera caerulea* L.) inhibits lipase activity and modulates the gut microbiota in high-fat diet-fed mice. *Molecules* **2022**, *27*, 4731. [[CrossRef](#)]
12. Lee, C.H.; Chen, K.T.; Lin, J.A.; Chen, Y.T.; Chen, Y.A.; Wu, J.T.; Hsieh, C.W. Recent advances in processing technology to reduce 5-hydroxymethylfurfural in foods. *Trends Food Sci. Technol.* **2019**, *93*, 271–280. [[CrossRef](#)]
13. Tomruk, D.; Devseren, E.; Koç, M.; Ocağ, Ö.Ö.; Karataş, H.; Kaymak-Ertekin, F. Developing a household vacuum cooking equipment, testing its performance on strawberry jam production and its comparison with atmospheric cooking. *Agron. Res.* **2016**, *14*, 1475–1487.
14. Kuşçu, A.; Bulantekin, Ö. Determination of phenolics, organic acids, minerals and volatile compounds of jujube (*Ziziphus jujuba* miller) jam produced by under vacuum evaporation compared with open pan method. *J. Food Meas. Charact.* **2021**, *15*, 1127–1138. [[CrossRef](#)]
15. Korus, A.; Jaworska, G.; Bernaś, E.; Juszczak, L. Characteristics of physico-chemical properties of bilberry (*Vaccinium myrtillus* L.) jams with added herbs. *J. Food Sci. Technol.* **2015**, *52*, 2815–2823. [[CrossRef](#)]
16. Gao, X.; Ohlander, M.; Jeppsson, N.; Björk, L.; Trajkovski, V. Changes in antioxidant effects and their relationship to phytonutrients in fruits of sea buckthorn (*Hippophae rhamnoides* L.) during maturation. *J. Agric. Food Chem.* **2000**, *48*, 1485–1490. [[CrossRef](#)]
17. Goiffon, J.P.; Mouly, P.P.; Gaydou, E.M. Anthocyanic pigment determination in red fruit juices, concentrated juices and syrups using liquid chromatography. *Anal. Chim. Acta* **1999**, *382*, 39–50. [[CrossRef](#)]
18. Yen, G.C.; Chen, H.Y. Antioxidant activity of various tea extracts in relation to their antimutagenicity. *J. Agric. Food Chem.* **1995**, *43*, 27–32. [[CrossRef](#)]
19. Re, R.; Pellegrini, N.; Proteggente, A.; Pannala, A.; Yang, M.; Rice-Evans, C. Antioxidant activity applying an improved ABTS radical cation decolorization assay. *Free Radic. Biol. Med.* **1999**, *26*, 1231–1237. [[CrossRef](#)]
20. Kruszewski, B.; Zawada, K.; Karpiński, P. Impact of high-pressure homogenization parameters on physicochemical characteristics, bioactive compounds content, and antioxidant capacity of blackcurrant juice. *Molecules* **2021**, *26*, 1802. [[CrossRef](#)]
21. Rada-Mendoza, M.; Olano, A.; Villamiel, M. Determination of hydroxymethylfurfural in commercial jams and in fruit-based infant foods. *Food Chem.* **2002**, *79*, 513–516. [[CrossRef](#)]
22. Grobelna, A.; Kalisz, S.; Kieliszek, M. Effect of processing methods and storage time on the content of bioactive compounds in blue honeysuckle berry purees. *Agronomy* **2019**, *9*, 860. [[CrossRef](#)]
23. Kuşçu, A.; Bulantekin, Ö. The effects of production methods and storage on the chemical constituents of apple pekmez. *J. Food Sci. Technol.* **2016**, *53*, 3083–3092. [[CrossRef](#)]
24. Devseren, E.; Okut, D.; Koç, M.; Karataş, H.; Kaymak-Ertekin, F. Comparison of quality characteristics of tomato paste produced under atmospheric conditions and vacuum evaporations. *An. Acad. Bras. Cienc.* **2021**, *93*, e20200215. [[CrossRef](#)]
25. Auzanneau, N.; Weber, P.; Kosińska-Cagnazzo, A.; Andlauer, W. Bioactive compounds and antioxidant capacity of *Lonicera caerulea* berries: Comparison of seven cultivars over three harvesting years. *J. Food Compos. Anal.* **2018**, *66*, 81–89. [[CrossRef](#)]
26. Patras, A.; Brunton, N.P.; Tiwari, B.K.; Butler, F. Stability and degradation kinetics of bioactive compounds and colour in strawberry jam during storage. *Food Bioprocess Technol.* **2011**, *4*, 1245–1252. [[CrossRef](#)]
27. Volf, I.; Ignat, I.; Neamtu, M.; Popa, V.I. Thermal stability, antioxidant activity, and photo-oxidation of natural polyphenols. *Chem. Pap.* **2014**, *68*, 121–129. [[CrossRef](#)]
28. Mohdaly, A.A.A.; Roby, M.H.H.; Sultan, S.A.R.; Groß, E.; Smetanska, I. Potential of low cost agro-industrial wastes as a natural antioxidant on carcinogenic acrylamide formation in potato fried chips. *Molecules* **2022**, *27*, 7516. [[CrossRef](#)]
29. Kamiloglu, S.; Pasli, A.A.; Ozcelik, B.; Van Camp, J.; Capanoglu, E. Influence of different processing and storage conditions on in vitro bioaccessibility of polyphenols in black carrot jams and marmalades. *Food Chem.* **2015**, *186*, 74–82. [[CrossRef](#)]
30. Kopjar, M.; Ivana, Đ.; Piližota, V. HMF formation and colour change of bitter orange and sweet orange jams during storage. *Croat. J. Food Sci. Technol.* **2010**, *2*, 11–15.
31. Djaoudene, O.; Louaileche, H. Effect of storage time and temperature on the nutritional quality of commercial orange jam. *SDRP J. Food Sci. Technol.* **2017**, *1*, 78–84. [[CrossRef](#)]
32. Gorzelany, J.; Basara, O.; Kapusta, I.; Paweł, K.; Belcar, J. Evaluation of the chemical composition of selected varieties of *L. caerulea* var. *kamtschatica* and *L. caerulea* var. *emphyllocalyx*. *Molecules* **2023**, *28*, 2525. [[CrossRef](#)]
33. Kucharska, A.Z.; Sokół-Łętowska, A.; Oszmiański, J.; Piórecki, N.; Fecka, I. Iridoids, phenolic compounds and antioxidant activity of edible honeysuckle berries (*Lonicera caerulea* var. *kamtschatica* Sevast.). *Molecules* **2017**, *22*, 405. [[CrossRef](#)]
34. Wang, Y.; Zhu, J.; Meng, X.; Liu, S.; Mu, J.; Ning, C. Comparison of polyphenol, anthocyanin and antioxidant capacity in four varieties of *Lonicera caerulea* berry extracts. *Food Chem.* **2016**, *197*, 522–529. [[CrossRef](#)]
35. Patras, A.; Brunton, N.P.; O'Donnell, C.; Tiwari, B.K. Effect of thermal processing on anthocyanin stability in foods; mechanisms and kinetics of degradation. *Trends Food Sci. Technol.* **2010**, *21*, 3–11. [[CrossRef](#)]
36. Zhukovets, T.; Özcan, M.M. Determination of bioactive properties, phenolic compounds and mineral contents of boiled fruit juice types. *Iran. J. Chem. Chem. Eng.* **2021**, *40*, 2042–2048. [[CrossRef](#)]

37. Abdelazim Mohdaly, A.A.; Ramadan, M.F. Characteristics, composition and functional properties of seeds, seed cake and seed oil from different *Brassica carinata* genotypes. *Food Biosci.* **2022**, *48*, 100752. [[CrossRef](#)]
38. Diaconeasa, Z.; Iuhas, C.I.; Ayvaz, H.; Rugină, D.; Stanilă, A.; Dulf, F.; Bunea, A.; Socaci, S.A.; Socaciu, C.; Pinte, A. Phytochemical characterization of commercial processed blueberry, blackberry, blackcurrant, cranberry, and raspberry and their antioxidant activity. *Antioxidants* **2019**, *8*, 540. [[CrossRef](#)]

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