



# Article Effect of Various Drying Methods on Physicochemical and Bioactive Properties of Quince Fruit (*Cydonia oblonga* Mill.)

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Abstract: The quince (Cydonia oblonga Mill.), due to its valuable bioactive properties and high healthpromoting potential, is becoming more and more popular for the prevention of many free radical diseases. Due to the high hardness of the flesh and its bitterness and astringency, quinces are rarely eaten in the form of fresh fruit, and much more often in the form of various preserves, or in the form of dried additives, e.g., to the tea. Heat treatment (including drying) affects not only the content of bioactive compounds, but also the antioxidant activity and organoleptic characteristics. Therefore, this study examined the physicochemical properties of quinces (including the content of dry matter, soluble solids (°Brix), water activity ( $a_w$ ), pH, total acidity and color changes (in the  $L^*a^*b^*$  space)), fresh and dried by various methods, i.e., freeze-drying and convection at 50 °C and 70 °C. In addition, the effect of various drying conditions on the content of selected bioactive compounds, i.e., tannins, carotenoids, flavonoids, phenolic acids and total polyphenols, was assessed, as well as the antioxidant properties of fresh quinces and quinces dried under different conditions. Based on the research, it can be concluded that the applied processes of the dehydration of quinces significantly changed both the physicochemical properties and the content of biologically active ingredients and antioxidant properties, while both fresh and dried fruit provide nutritionally valuable bioactive ingredients and show high antioxidant potential. Considering the great taste and bioactive qualities of the common quince, introducing it to the daily diet, whether in a traditional form (dried fruit, fruit preserves) or in the form of dietary supplements, can be an important element in the prevention of many civilization diseases.

**Keywords:** quince fruit; physicochemical properties; bioactive compound content; antioxidant activity

# 1. Introduction

The quince (*Cydonia oblonga* Mill.) is one of the oldest fruit species in the world. It comes from Southeast Asia but is widespread on many continents, including Europe, especially southern Europe [1]. The quince (*Cydonia oblonga* Mill.) belongs to the class of angiosperms, the rose family. It received its generic name thanks to the Romans, who, after discovering the tree in the Cretan city of Kydon, gave it the name malum cydonium, or apple of Kydon [2].



Citation: Najman, K.; Adrian, S.; Hallmann, E.; Sadowska, A.; Buczak, K.; Waszkiewicz-Robak, B.; Szterk, A. Effect of Various Drying Methods on Physicochemical and Bioactive Properties of Quince Fruit (*Cydonia oblonga* Mill.). *Agriculture* **2023**, *13*, 446. https://doi.org/10.3390/ agriculture13020446

Academic Editor: Quan-Sheng Chen

Received: 5 January 2023 Revised: 8 February 2023 Accepted: 12 February 2023 Published: 14 February 2023



**Copyright:** © 2023 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). In Polish climatic conditions, the quince reaches an average height of 3 m [3]. The flowers resemble apple blossoms in appearance but they do not grow in bunches, only singly, and are 4–5 cm in diameter on a stalk with egg-shaped bracts, having five white or pink petals about 2 cm long. Its flowering period is at the end of May, beginning of June, after which the flowers form fruit similar to apples or pears, depending on the variety [3–6]. The vast majority of quince varieties do not self-pollinate with their own pollen, so, in order to maintain the natural cycle of fruiting, it is recommended to plant 2–3 flowering trees at the same time [4]. In Polish conditions, the fruit ripen relatively late, i.e., at the turn of September and even the end of October [3,7]. Ripe fruit are yellow and have an aromatic smell, and their lemon-yellow skin is covered with visible but easily removable moss [5,8,9]. Depending on the variety, the fruit reach a diameter of 3–5 cm and a weight of 100–200 g, and some varieties even up to 1 kg [3,7,10,11].

Despite very favorable natural conditions and modest requirements for the use of agricultural techniques in the cultivation of the quince, it is difficult to explain the fact that it is very poorly represented in the fruit industry. In 2020, worldwide realized quince production amounted to almost 700,000 t with Turkey and China growing a combined 43% of the world total. Over the past few years, there has been a trend of increasing quince production in the world, and in 2003–2012 it was about 469,325 t [12,13].

The medicinal properties of the quince have been known for centuries [5]. In traditional folk medicine, various ailments have been treated with roots, fruits, leaves and seeds of this plant., e.g., fruit seeds were used to treat gastrointestinal disorders such as constipation and diarrhea, quince leaf decoctions were used to treat nervousness, insomnia, cough and fever and the fruit itself helped in disorders, respiratory diseases, urinary tract diseases, peptic ulcer disease or diabetes [5,14]. Research conducted in recent years has confirmed the high therapeutic potential of this plant [15-17]. According to the literature, the aqueous extract of Cydonia oblonga Mill. showed hypolipidemic, (improving the lipid profile by lowering the level of total cholesterol, triglycerides and the low-density lipoprotein (LDL) cholesterol fraction while increasing the level of the high-density lipoprotein (HDL) cholesterol fraction in the blood serum), hepatoprotective (lowering the levels of liver enzymes ALT, AST and ALP in the serum) and renoprotective (lowering the concentration of urea and creatinine) effects in diabetic rats during an experiment lasting 6 weeks [18]. In the literature, the antiatherosclerotic, hypoglycemic [19], antioxidant [20,21] and anti-inflammatory [22,23] properties of *Cydnia oblonga* fruit are also known. Studies conducted by Carvalho et al. (2010) [24] also showed the inhibition of the growth of human colon cancer cells by a quince leaf extract, as well as the antiproliferative effect of a quince seed extract on kidney cancer cells, thus confirming, also by other authors, the anticancer properties of these fruit [25]. In other studies, the antibacterial effect of ethanol extract of quince seeds (Cydonia oblonga) against such bacteria as Staphylococcus aureus, Staphylococcus epidermidis and Escherichia coli was demonstrated, with a better effect on gram-positive than gram-negative bacteria [26].

The wide health benefits of quince consumption are attributed to the bioactive components found in its fruit, primarily those with antioxidant properties [8,27–31]. These fruit are characterized by a high content of polyphenols (the highest concentration of which is found in the skin of the fruit) and carotenoids, showing high antioxidant activity [32,33]. The quince is a very good source of vitamin C [34], and it also provides valuable minerals, e.g., potassium, calcium, magnesium or sodium, organic acids and amino acids [34–39].

Taking into account the valuable nutritional properties of the quince as well as its pro-health potential, this plant is gaining more and more popularity both as a subject of research by scientists and a promising biopharmaceutical, possibly to be used in the prevention of many free radical diseases [40–44]. It is also known among consumers, using a variety of quince products available on the market or as a raw material for processing at home [17].

Quinces, due to their very hard flesh and sour and tart taste, are not suitable for direct consumption (raw), but thanks to the content of compounds such as tannins, sugars, pectins and aroma compounds, these fruits are very widely used in the preparation of preserves,

such as jams, plum jam, jellies, juices, syrups and dried products [5,33,45–49]. They are also appreciated as a raw material in the food alcohol industry, e.g., for the production of tinctures (as their main ingredient) or a flavoring additive to stronger alcohols [50]. Due to the high taste and aroma similarity to lemon fruit [51], these fruit are very often used as an addition to cakes, desserts or drinks, e.g., for teas, and they are also often a component of their mixtures in the form of dried leaves and pieces of fruit [52–56]. According to the literature, infusions with the addition of quince show, e.g., a strong antioxidant effect and also protect erythrocytes from oxidative damage [40].

Heat treatment (including drying) affects not only the content of bioactive compounds, such as polyphenols, carotenoids or vitamins, leading to their losses and thus to a reduction in antioxidant activity to an extent that depends on the type of drying process used [57], but also changes organoleptic characteristics, affecting the texture, color, smell and taste of the products, often leading to their deterioration [58]. According to the literature, both the total content of phenolic compounds (as bioactive components with strong antioxidant properties) and the total antioxidant activity decreased depending on the type of -drying process used (i.e., air, sun, oven and microwave drying), with the greatest losses found in air-dried fruit [56,59].

Convection drying is one of the oldest, widely used methods of producing dried fruit in the food industry. The most important disadvantages of convection drying are nutrient losses, color changes and long process times. Dehydration by the sublimation of water from the frozen product is the basis of freeze-drying. Since there is no liquid water in the product structure and a low temperature is required for freeze-drying, deterioration and microbial activity are mostly inhibited or delayed, resulting in a final product that is microbiologically safe. Among the positive aspects of carrying out this process is the protection in a vacuum of substances that are susceptible to oxidation. As a result, the loss of heat-sensitive bioactive components is minimized. There is also no change in the color of the product. One of the few disadvantages of freeze-drying is its cost, higher than that of traditional drying by means of high temperature [59–61].

There are numerous studies in the literature comparing freeze-drying and convection drying methods, but relatively few works on quince drying [60,62–64]. The available literature lacks data on the effect of various drying conditions of quinces on the physical properties and characteristics (including dry matter content, water activity ( $a_w$ ), pH, total acidity, soluble solids content (°Brix), color parameters in the  $L^*a^*b^*$  color space)), bioactive properties, i.e., the content of selected biologically active ingredients (including tannins, carotenoids, flavonoids, phenolic acids, total polyphenols) and antioxidant activity of quinces (*Cydonia oblonga* Mill.) dried by freeze-drying and convection. The research carried out allows for the selection of appropriate methods of drying quince fruit in order to ensure and maintain the highest possible content of thermolabile bioactive ingredients with high antioxidant properties, both for consumers preparing dried fruit at home and for food producers using quince or their processing products in the design of functional foods.

#### 2. Materials and Methods

#### 2.1. Materials

The research material consisted of ripe fruit of common quinces (*Cydonia oblonga* Mill.) from an organic farm located in the province of Świętokrzyskie, collected in autumn 2021. The fresh fruit were stored in a cold store at 10 °C and 85% relative air humidity for 24 h. A total of 20 pieces of fruit of similar weight ( $215.0 \pm 15.11$  g on average) were selected for this study, which were washed, dried, cut into thin slices of 3–4 mm thick and divided into 4 parts: one part was intended for the assessment of bioactive and physicochemical properties of the fresh fruit (Fresh), the other 3 parts were dried, i.e., freeze-drying (Freeze-Dried) and convective drying at 70 °C (Conv-70) and 50 °C (Conv-50). The fruit samples intended for freeze drying were frozen in liquid nitrogen and then freeze-dried (Alpha 1-4 LSC, Martin Christ GmbH, Osterode am Harz, Germany) at a pressure in the chamber of 10 Pa, temperature in the drying chamber of -50 °C and shelf temperature of 21 °C for

72 h. Fruit samples intended for convection drying were placed on trays and dried in a laboratory dryer with air circulation (SUP-200G Wamed, Warsaw, Poland) at a temperature of 70 °C for 24 h and at 50 °C for 48 h (respectively, for C-70 and C-50), to water activity  $a_w < 0.3$ . Freeze-dried and convectively dried research material was ground in a laboratory knife mill (Grindomix GM 200, Retsch GmbH, Haan, Germany), sealed in plastic containers and stored until testing. Figure 1 shows the scheme of the study.

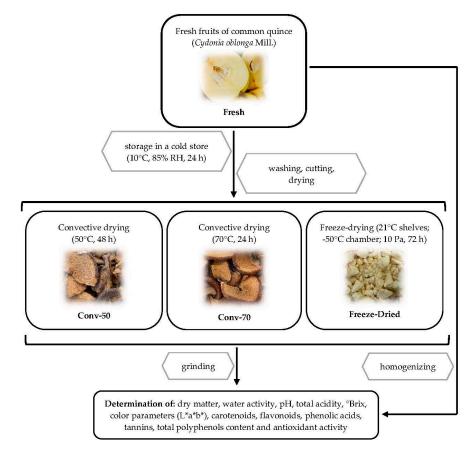


Figure 1. The flowchart of experimental design.

# 2.2. Methods

Physicochemical properties

#### 2.2.1. Dry Matter Content

The dry matter content in the quince samples was determined gravimetrically, according to the AOAC method (2000) [65]. The vessels were weighed and cooled down, and then 5.0 g (with an accuracy of 0.0001 g) of the samples was weighed out on an analytical balance (AS 220/X, Radwag, Radom, Poland). The samples were dried in a laboratory dryer (SUP 200 W, Wamed, Warsaw, Poland) for 72 h at 105 °C. The dry matter content was calculated from the weight differences, and the result was expressed as a percentage (%).

#### 2.2.2. Water Activity $(a_w)$

Water activity (a<sub>w</sub>) was measured using a hand-held water activity meter with a temperature stabilizer, the AquaLab Water Activity Meter (Decagon Devices. Inc., Pullman, WA, USA).

# 2.2.3. pH

The pH measurement in the tested samples was carried out using the potentiometric method using a laboratory pH-meter probe (Elmetron CP-511, Elmetron G.P., Zabrze, Poland), at room temperature (20 °C).

#### 2.2.4. Total Acidity (TA)

Total acidity was determined using the titration method described in the Polish Standard (PN-EN 12147:2000) [66], consisting of neutralizing the acids present in the tested samples by titration with a 0.1 M NaOH (sodium hydroxide) solution (Sigma-Aldrich, Poznań, Poland) in the presence of phenolphthalein (Sigma-Aldrich, Poznań, Poland) as an indicator.

In order to perform the determination, 25 g of the test material was weighed (with an accuracy of 0.01 g) into 250 mL beakers on an analytical balance (AS 220/X, Radwag, Radom, Poland), 100 mL of distilled water was added, then it was heated to boiling, and, after cooling down, quantitatively transferred to a 250 mL volumetric flask, filled up to the mark with distilled water, closed and left for 15 min at room temperature (20 °C). The contents of the flask were filtered through a corrugated filter into a beaker, 10 mL of the solution was taken from the obtained filtrate, and 3 drops of phenolphthalein were added and titrated with a 0.1 M NaOH solution until the raspberry color persisted for at least 30 s. The total acidity (TA) was converted, in accordance with the requirements of the relevant standards, depending on the specificity of the tested product (for pome and stone fruits), into malic acid and expressed as g/100 g of the product.

# 2.2.5. Soluble Solids Content (°Brix)

Soluble solids content (°Brix) was measured with an Abbe refractometer (ORT-1, Kern & Sohn GmbH, Balingren-Ffrommern, Germany) using the refractometric method described in the Polish Standard PN-EN 12143:2000 [67], based on the measurement of the refraction of light in the tested material.

#### 2.2.6. Instrumental Color Measurement

The color of the tested quince samples was measured at room temperature (20  $^{\circ}$ C) using a colorimeter (Konica Minolta CR-400, Konica Minolta, BSP, Warsaw, Poland). The samples were placed in a glass measuring pan (60 mm in diameter) on the measuring head, and the results were read for 3 parameters of the colorimetric model of the CIE Lab color space (L-lightness, +a-red, –a-green, +b-yellow, –b-blue).

### Bioactive properties

# 2.2.7. The Content of Selected Carotenoids

Quantitative and qualitative analysis of carotenoids in the tested quince samples was carried out using the HPLC method according to Ponder et al. (2021) [68]. In order to perform the extraction of carotenoids, 30.0 mg (with an accuracy of 0.1 mg) of the tested samples were weighed on an analytical balance (AS 220/X, Radwag, Radom, Poland) into 10 mL glass tubes, then 10 mg of MgCO<sub>3</sub> (magnesium carbonate) (Sigma-Aldrich, Poznań, Poland) and 5.0 mL of acetone (Sigma-Aldrich, Poznań, Poland) were added. The samples were vortexed (Wizard Advanced IR Vortex Mixer, VELP Scientifica Srl, Usmate (MB), Italy) for 60 s at 2000 rpm, then incubated in an ultrasonic bath (Bandelin Sonorex RK 255, BANDELIN Electronic, Berlin, Germany) (35 kHz, 20 °C, 15 min), centrifuged in a refrigerated centrifuge (MPW-380 R, MPW Med. Instruments, Poland, Warsaw) for 15 min (4 °C, 10,000 rpm), and the obtained supernatant was transferred into HPLC-vials and used for determination.

For the determination of carotenoids, the Shimadzu HPLC set (Shim-pol, Warsaw, Poland) was used, which included two LC-20AD pumps, a CMB-20A set controller, an SIL-20AC autosampler, a CTD-20A and a Max-RP 80A column ( $4.6 \times 250$  mm). The mobile phase was prepared from a mixture of acetone and n-hexane (5:95) and used as a

gradient phase. The flow time was 1.5 mL/min; the wavelength range was 445–480 nm. For the qualitative identification of carotenoid compounds, external standards of substances (Sigma-Aldrich, Poznań, Poland) with a purity of 99.9% were used. For the quantitative identification of individual carotenoids, previously prepared standard curves for standard substances were used, and after taking into account the dilutions used, the content of selected carotenoids (lutein, zeaxanthin, chlorophyll a, b-chlorophyll b and  $\beta$ -carotene) was expressed as mg/100 g of the product. The pictures of the chromatographic spectra of the carotenoids HPLC analysis are presented in Figure S5 (Fresh), Figure S6 (Conv-50), Figure S7 (Conv-70) and Figure S8 (Freeze-Dried) (Supplementary Materials).

#### 2.2.8. The Content of Selected Flavonoids and Phenolic Acids

Quantitative and qualitative analysis of selected flavonoids and phenolic acids in the tested quince samples was carried out by HPLC according to Hallmann et al. (2017) [69]. In order to perform the HPLC extraction of phenolic compounds, 100.0 mg of the tested samples were weighed into plastic test tubes with a capacity of 10 mL on an analytical balance (AS 220/X, Radwag, Radom, Poland), with an accuracy of 0.01 g, followed by the addition of 5.0 mL of 80% methanol (Sigma-Aldrich, Poznań, Poland). The extracts were shaken for 60 s at 2000 rpm using a vortex shaker (Wizard Advanced IR Vortex Mixer, VELP Scientifica Srl, Usmate (MB), Italy), then incubated in an ultrasonic bath (Bandelin Sonorex RK 255, BANDELIN Electronic, Berlin, Germany) (35 kHz, 30 °C, 5 min), centrifuged in a refrigerated centrifuge (MPW-380 R, MPW Med. Instruments, Poland, Warsaw) for 15 min (4 °C, 10,000 rpm) and 1 mL of the obtained supernatant was collected in HPLC-vials and analysed.

The content of selected phenolic compounds was determined using the Shimadzu HPLC set (USA Manufacturing Inc., USA), consisting of 2 LC-20AD pumps, CMB-20A and CTD-20AC system controllers, an SIL-20AC autosampler and an SPD-20AV UV/VIS. Phenolic compounds were identified and separated on a Synergi Fusion-RP 80i ( $250 \times 4.60$  mm) chromatography column using a two-phase flow gradient: acetonitrile/deionized water (55% and 10%) at pH 3.00 for 38 min at flow rate of 1.0 mL/min and detection at wavelengths of 250–370 nm. For the qualitative identification of phenolic compounds, external standards of substances (Sigma-Aldrich, Poznań, Poland) with a purity of 99.9% were used. The content of selected phenolic compounds (phenolic acids: gallic, chlorogenic, p-coumaric, ferulic, coffee; flavonoids: catechin, epigallocatechin, rutoside-3-O-quercetin, quercetin) in the quince samples was calculated using the prepared standard curves for standard substances, and the results were expressed as mg/100 g of product. The pictures of the chromatographic spectra of the polyphenols HPLC analysis are presented in Figure S1 (Fresh), Figure S2 (Conv-50), Figure S3 (Conv-70) and Figure S4 (Freeze-dried) (Supplementary Materials).

#### 2.2.9. Tannin Content

Determination of the content of tannins in the tested quince samples was carried out using the titration and weight method according to Ciszewska et al. (1975) [70], consisting of precipitating protein substances from the infusion, and then, after adding the appropriate amount of potassium iodide (at a concentration of 10%) (KI, Sigma-Aldrich, Poznań, Poland), titration of the secreted tannins by sodium thiosulfate solution (0.05 M Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>) (Sigma-Aldrich, Poznań, Poland) with starch as an indicator.

In order to perform the determination, 6.0 g (with an accuracy of 0.001 g) of the tested quince samples were weighed into 400 mL beakers on an analytical balance (AS 220/X, Radwag, Radom, Poland), then 250 mL of boiling distilled water was added, left for 10 min under cover, and then filtered through a corrugated filter into a 250 mL measuring cylinder. After filtration, 175 mL of the filtrate was transferred into a glass beaker and heated to boiling, then 20 mL of a 4% solution of copper (II) acetate (CuSO<sub>4</sub>) (Sigma-Aldrich, Poznań, Poland) was added, quantitatively transferred to a 200 mL volumetric flask, cooled, topped up with distilled water to the mark and again filtered through a fluted filter. Then, 100 mL

of the obtained filtrate was collected into a 200 mL conical flask, 25 mL of 50% acetic acid (Sigma-Aldrich, Poznań, Poland), 20 mL of 10% potassium iodide solution and 1.5 mL of 2% aqueous starch solution (indicator) were added and titrated with a 0.05 M Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solution for a minimum of 30 s of color change of the solution. The content of tannins was expressed as g/100 g of the product.

# 2.2.10. Preparation of Extracts for Determination of Total Polyphenol Content and Antioxidant Activity

In order to extract phenolic compounds, 500.0 mg of tested quince samples were weighed into 50 mL plastic Falcone tubes on an analytical balance (AS 220/X, Radwag, Radom, Poland), with an accuracy of 0.01 g, and then 40 mL of distilled water was added. The extracts were shaken for 60 s at 2000 rpm. using a vortex shaker (Wizard Advanced IR Vortex Mixer, VELP Scientifica Srl, Usmate (MB), Italy), then incubated in a shaking incubator (IKA KS 4000i Control, IKA<sup>®</sup> Poland Ltd., Warsaw, Poland) for 60 min (60 °C, 200 rpm). After incubation, the extracts were vortexed for 60 s and then centrifuged in a refrigerated centrifuge (MPW-380 R, MPW Med. Instruments, Poland, Warsaw) for 15 min (4 °C, 10,000 rpm). The supernatant obtained in this way was used to determine the total polyphenol content and antioxidant activity.

#### 2.2.11. Total Polyphenol Content

The content of total polyphenolic compounds in the tested quince samples was determined by the colorimetric spectrophotometric method according to Singleton and Rossi (1965) [71] using the Folin–Ciocalteu reagent (Sigma-Aldrich, Poznań, Poland). In order to carry out the determination, 1.0 mL of appropriately diluted solutions of extracts of the tested samples were taken to 50.0 mL volumetric flasks, then 2.5 mL of Folin–Ciocalteu reagent and 5.0 mL of sodium carbonate with a concentration of 20% (Na<sub>2</sub>CO<sub>3</sub>, Sigma-Aldrich, Poznań, Poland) were added, supplemented with distilled water to the mark, stoppered, gently mixed, and then incubated for 60 min at room temperature (20 °C), protected from light. After incubation, the absorbance of the solutions was measured at  $\lambda$ = 750 nm using a spectrophotometer (UV/Vis UV-6100A, Metash Instruments Co., Ltd., Shanghai, China). The results, based on the obtained absorbance measurements, taking into account the dilution schemes used during the analysis, were calculated on the basis of the standard curve for gallic acid (Sigma-Aldrich, Poznań, Poland) as a standard and expressed as mg GAE/100 g of product (GAE—gallicacid equivalent).

#### 2.2.12. Antioxidant Activity

Antioxidant activity in the tested quince samples was determined by the colorimetric spectrophotometric method according to Re et al. (1999) [72] using the cation radical ABTS<sup>+•</sup> (2,2'-azino-bis 3-ethylbenzothiazolin-6-sulfonic acid) (Sigma-Aldrich, Poznań, Poland). In order to carry out the determination, 0.5–1.5 mL of appropriately diluted solutions of extracts were collected into 10 mL glass tubes, 3.0 mL of a solution of radical cations ABTS<sup>+•</sup> in PBS (Phosphate Buffer Solution, Sigma-Aldrich, Poznań, Poland) was added (with a precisely defined content of radicals, giving at  $\lambda$  = 734 nm absorbance 0.700 ± 0.02), it was stirred for 5 s at 2000 rpm on a vortex shaker (Wizard Advanced IR Vortex Mixer, VELP Scientifica Srl, Usmate (MB), Italy), incubated for exactly 6 min at room temperature (20 °C), and then the absorbance of the solutions was measured at  $\lambda$  = 734 nm, using a spectrophotometer (UV/Vis UV-6100A, Metash Instruments Co., Ltd., Shanghai, China). The results, based on the obtained absorbance measurements, taking into account the applied dilution schemes, were calculated on the basis of the standard curve for Trolox (Sigma-Aldrich, Poznań, Poland) as a standard and expressed as  $\mu$ M TEAC/100 g of product (TEACTrolox-equivalent antioxidant capacity).

#### 2.2.13. Statistical Analysis

Measurements and determinations in the research material were performed in 3–6 independent repetitions. The results are presented in tables as mean values  $\pm$  standard deviation (SD). Analysis of variance (ANOVA) was performed using the Statistica 13.0 software (Tibco Software Inc., Palo Alto, CA, USA), and the differences between the groups were determined by a Duncan's test at the assumed significance level of *p* < 0.05.

#### 3. Results and Discussion

#### 3.1. Physicochemical Properties of Quince Fruit Fresh and Dried by Various Methods

This study evaluated parameters and physical properties such as dry matter, water activity  $(a_w)$ , pH, total acidity and soluble solids content (°Brix) in quinces fresh and dried by various methods. The results are presented in Table 1.

Parameter	Fresh	Conv-50	Conv-70	Freeze-Dried
Dry matter (%)	$18.60\pm0.16\ ^{a}$	$86.99\pm0.11~^{\rm b}$	$87.20 \pm 0.39$ <sup>b</sup>	$98.58\pm0.18\ ^{\mathrm{c}}$
Water activity (a <sub>w</sub> )	$0.9857 \pm 0.00$ <sup>d</sup>	$0.2633 \pm 0.00$ <sup>c</sup>	$0.2133 \pm 0.00$ <sup>b</sup>	$0.1352\pm0.00$ $^\circ$
pН	$4.19\pm0.03$	-	-	-
Total acidity (g/100 g)	$0.26\pm0.00~^{\rm a}$	$1.65\pm0.01~^{\rm b}$	$1.83\pm0.04$ c	$1.87\pm0.01~^{\rm c}$
°Brix (%)	$12.50\pm0.50$	-	-	-

Table 1. Physicochemical properties of fresh and dried quince.

Values are means  $\pm$  standard deviation (n = 3). <sup>a–d</sup>—different letters in the same line are significantly different (Duncan's test, *p* < 0.05). Fresh—fresh fruit; Conv-50—convection-dried fruit at 50 °C; Conv-70—convection-dried fruit at 70 °C; Freeze-Dried—freeze-dried fruit.

The content of the dry matter in the fresh fruit was about 19% and in the dried fruit between 87% and 99% (respectively, for convection and freeze-dried methods). The observed trends are confirmed in studies by other authors on the comparison of the quality of fruit and fruit powders dried using different methods [73,74].

As can be seen from the data presented in Table 1, the highest water activity was found in fresh fruit, and it was more than four times lower (due to dehydration of products) in dried fruit. The average water activity in dried fruit was  $0.2039 \pm 0.06$ , with different drying methods significantly (p < 0.05) affecting the  $a_w$  of these products. The lowest  $a_w$ was found in freeze-dried fruit and it was higher in convection-dried fruit, where drying at a higher temperature resulted in lower a<sub>w</sub> values. Water activity in the product is a decisive feature for biological processes and the development of microorganisms. It is assumed that microorganisms such as bacteria, yeasts and molds cannot develop in food whose  $a_w$ is lower than 0.6 and such food can be considered microbiologically safe, while ensuring its greater durability [73]. No data on the effect of various methods of quince drying on water activity were found in the literature. However, according to the literature, various methods of dehydration of fruit and vegetable raw materials or their products, e.g., fruit powders, affect the water activity in the final product [73,74]. In addition, low a<sub>w</sub> promotes greater stability of water-soluble substances, e.g., vitamin C, as well as other bioactive ingredients, such as carotenoids, flavonoids or other food ingredients with antioxidant properties [73,74]. The lowest  $a_w$  in our research was found in freeze-dried quince fruit; however, it should be remembered that, due to their high hygroscopicity, they require proper storage. High water activity and high humidity of plant material are also conducive to enzymatic and non-enzymatic browning reactions, i.e., Maillard reactions, which may result in changes in the taste, aroma and color of products [75,76], which was also observed in this research.

As a result of the conducted research, quince was characterized by an acidic pH (4.19  $\pm$  0.03), which was confirmed by literature data. Al.-Zughbi et al. (2022) [17] showed

the pH of fresh quince fruit to be 3.43. Other authors, studying, e.g., the chemical properties of five Spanish quince cultivars, reported a fruit pH of between 3.60 and 3.84 [10]. In contrast, Szychowski et al. (2014) [77], in research conducted on six varieties of quince, additionally harvested in two harvesting seasons, showed the pH in the fruit in the range from 3.96 to 4.09, which was similar to the pH for the fresh quince tested in this study. A decrease in pH was also observed during the ripening of the quinces during the 5-month storage period (from 4.92 to 3.07), most likely due to the accumulation of sugars in the fruit [78]. According to the literature, the pH of fresh quinces may vary depending on the variety, growing and harvesting conditions, and the degree of maturity [10,17,77].

High fruit pH usually corresponds to low total acidity [33,45], which was also reflected in the conducted research (Table 1). The lowest total acidity in terms of malic acid was found in fresh quince fruit ( $0.26 \pm 0.00 \text{ g}/100 \text{ g}$ ), while dried ones showed almost seven times higher values of this parameter (average  $1.78 \pm 0.11 \text{ g}/100 \text{ g}$ ), with significantly (p < 0.05) the highest in freeze-dried quinces ( $1.87 \pm 0.01 \text{ g}/100 \text{ g}$ ). Szychowski et al. (2014) [77], in a study comparing six quince varieties, showed acidity in the range of 0.40 g/100 g to 0.55 g/100 g, and similar values were also obtained by Sharma et al. (2011) [34]. However, Rodríguez-Guisado et al. (2009) [10] obtained much higher values for different varieties of Spanish quinces (from 0.47 to 0.79 g/100 g). The total acidity in the fresh quinces studied in this work was several times lower than in the studies of other authors, which, according to the literature, could be caused by the diversity of quince cultivars and genotypes, growing conditions, harvesting, agricultural practices or the degree of fruit ripeness [10,27,77].

This study also examined the content of soluble solids (°Brix) in fresh quinces (Table 1). The most similar values for this parameter were shown by Rodríguez-Guisado et al. (2009) [10] and Szychowski et al. (2014) [77], who in various quince varieties found the content of soluble solids in the range of 11.57–14.70% and 15.10–17.20%, respectively. Lower values of °Brix (5.28–9.54%) in nine quince cultivars were shown by Legua et al. (2013) [27]. The authors explained the differences in the obtained results with genetic and environmental factors as well as the degree of ripeness of the examined fruit, in which sugars naturally accumulate during storage and ripening, and their profile changes. According to the literature, the average content of the reducing sugars in the fruit of various quince cultivars ranged from 566 to 785 mg/100 g d.m., with the highest share of fructose (416–584 mg/100 g d.m.) and significantly lower glucose (82.5–112 mg/100 g d.m.) [66]. Similar values were also obtained by Rodríguez-Guisado et al. (2009) [10], who, comparing five cultivars of quince, showed that reducing sugars accounted for approx. 85% of all sugars in quinces. The content of all reducing sugars in juices from different quince cultivars ranged from 11.67 to 16.08%, with the highest share of fructose (6.70–10.89%), lower glucose (4.08–5.61%)) and sucrose (1.51–2.41%) and the lowest for maltose (0.31–0.42%).

According to the literature, the generally high content of soluble solids and low total acidity indicate a high maturity rate of fresh fruit and their suitability for direct consumption [77]. The results for these parameters obtained in our research were similar to those of other authors, and therefore the fresh fruit studied in this work could be intended for direct consumption. However, much more often, due to the high astringency, bitterness and semi-hard pulp, quince fruit are used for various types of fruit preserves [10,17,27,77]. In terms of processing, they are a world-renowned raw material for processing into marmalades, preserves, jams and jellies, as well as an addition to cakes, desserts or drinks [28,45,47,49,52–56]. Moreover, in Poland, fruit harvested in autumn are willingly used in industrial and home fruit processing, while in dried form they are used in the production of compotes or as an addition to tea [78,79].

In the quinces, fresh and dried by various methods, significant differences in color parameters were found for each of the analyzed parameters in the  $L^*a^*b^*$  color space, and the obtained results are presented in Table 2.

Color Parameter	Fresh	Conv-50	Conv-70	Freeze-Dried
L* (lightness)	$82.11\pm3.48~^{\rm c}$	$59.21\pm1.31~^{\rm b}$	$56.31\pm1.46~^{\rm a}$	$91.72\pm0.22~^{\rm d}$
a* (redness)	$11.54\pm0.59~^{\rm d}$	$9.02\pm0.86^{\ c}$	$8.53\pm0.18~^{\rm b}$	$-1.37\pm0.16$ $^{\rm a}$
b* (yellowness)	$58.76\pm2.01~^{\rm d}$	$35.27\pm0.50~^{\rm c}$	$34.55\pm0.10^{\text{ b}}$	$33.12\pm0.59~^{\rm a}$

**Table 2.** Color parameters in  $L^*a^*b^*$  space of fresh and dried quince.

Values are means  $\pm$  standard deviation (n = 3). <sup>a–d</sup>—different letters in the same line are significantly different (Duncan's test, *p* < 0.05). Fresh—fresh fruit; Conv-50—convection-dried fruit at 50 °C; Conv-70—convection-dried fruit at 70 °C; Freeze-Dried—freeze-dried fruit.

Comparing the color of fresh and dried quince samples, the greatest differences were found in the case of the  $L^*$  parameter, which is the average for all analyzed samples of 72.34  $\pm$  15.76, with significant (p < 0.05) differences in this parameter. The highest value of the  $L^*$  color parameter was found in the freeze-dried quinces (91.72  $\pm$  0.22), which means that these samples were the lightest among all the analyzed samples. The freeze-dried quinces were about 10.5% brighter than the fresh quinces, for which the brightness parameter was 82.11  $\pm$  3.48. Convection-dried quince samples had a significantly darker color, with the lowest values for this parameter in fruit dried by convection at 70 °C (56.31  $\pm$  1.46).

The  $a^*$  parameter in the  $L^*a^*b^*$  system, referring to the tones of red (+ $a^*$ ) and green ( $-a^*$ ), was significantly different in fresh and dried quince samples, with the lowest value recorded in a freeze-dried quince sample ( $-1.37 \pm 0.16$ ), which means that these samples were characterized by a greater color shift towards shades of green. The remaining fruit samples showed positive values of the  $a^*$  parameter, which means that their color was shifted towards shades of red. Convectively dried fruit were characterized by a significantly (p < 0.05) higher redness (average 8.78 ± 0.62) than freeze-dried fruit, with a higher red color saturation found in Conv-50 than in Conv-70. However, the highest value of the  $a^*$  parameter was recorded for fresh fruit (11.54 ± 0.59), which means that they were characterized by the largest color shift towards shades of red among all analyzed quince fruit samples.

All the tested samples of quince, both fresh and dried, were characterized by positive values of the  $b^*$  parameter in the  $L^*a^*b^*$  color space (the average value of this parameter was 40.42 ± 11.13), which means that they showed color shift towards yellow. However, significant (p < 0.05) differences were found in the intensity and saturation of this color in individual fruit samples, with the highest values in fresh fruit (58.76 ± 2.01), lower in convectively dried fruit (average 34.91 ± 0.51) and the lowest in lyophilized (33.12 ± 059).

Changes in the color of quince fruit, especially  $L^*$  and  $a^*$  color parameters, during convection drying, may be explained by the oxidation of dyes, due to a longer exposure to oxygen and higher drying temperature and by the production of dark substances during this process, i.e., melanoidins, responsible for the brown color of quince fruit [75,76]. Melanoidins are formed in the late stages of the Maillard reaction from sugars and amino acids, as polymers, polycondensation products of fructans and/or pyrroles. The speed and intensity of browning dried fruit during heat treatment is closely related to temperature, relative humidity, water activity and processing time. Maillard reactions are faster at higher temperatures, so an increase in temperature accelerates and intensifies the browning processes. Conversely, the higher the relative humidity and an aw higher than 0.7, the slower the rate and intensity of browning. The intensity of the Maillard reaction reaches a maximum in aw 0.3–0.7, depending on the type of product. Additionally, along with extending the heat treatment time, the browning intensity of dried food also increases [76,80,81]. In addition, during the contact of quince fruit flesh with atmospheric air, an enzymatic darkening reaction occurs. Ripe quince fruit have a golden-yellowish color. After cutting them, they darken very quickly, which is related to the oxidation of polyphenolic compounds contained in the fruit flesh. The high content of chlorogenic acid and the high activity of polyphenol oxidase enzymes make the flesh of the quince darken very quickly

after grinding (changes in  $L^*$  and  $a^*$  color parameters) [82]. Therefore, the brightness of fruit subjected to sublimation drying, at low pressure, is higher as compared to raw and convection-dried fruit, while the red color is less intense.

Therefore, it is very important, from the point of view of organoleptic evaluation and the overall quality of dried quince products, to determine the effect of various drying methods on color parameters. Unfortunately, the literature lacks data on the impact of various types of drying of these fruit on changes in the color profile. Research on the influence of drying methods on the color of other fruit, e.g., the chokeberry [73], strawberry [83] powders or dried mango seeds [84], confirm that freeze-dried fruit or fruit powders are characterized by a much brighter color (higher  $L^*$  values).

# 3.2. Bioactive Properties of Fresh and Dried by Various Methods Quince

This study examined the effect of convection drying (50 °C and 70 °C) and freezedrying on the content of selected bioactive ingredients, i.e., carotenoids, phenolic compounds and tannins, and the antioxidant potential in quinces, and the obtained results are presented in Tables 3-6.

Carotenoids	Fresh	Conv-50	Conv-70	Freeze-Dried
Lutein (mg/100 g)	$3.55\pm0.06$ $^{\rm a}$	$9.62\pm0.15^{\text{ c}}$	$8.88\pm0.19~^{b}$	$13.57\pm0.21~^{\rm d}$
Zeaxanthin (mg/100 g)	$1.39\pm0.00~^{a}$	$5.56\pm0.00~^{\rm b}$	$5.56\pm0.00~^{b}$	$5.57\pm0.00~^{\rm b}$
Chlorophyll a (mg/100 g)	$14.22\pm0.13~^{\rm a}$	$39.31\pm0.46~^{\rm c}$	$35.72 \pm 0.24$ <sup>b</sup>	$57.88 \pm 0.22$ <sup>d</sup>
Chlorophyll b (mg/100 g)	$12.22\pm0.16~^{\rm a}$	$36.68 \pm 1.05$ <sup>c</sup>	$33.40 \pm 0.62$ <sup>b</sup>	$49.23 \pm 0.37$ <sup>d</sup>
β-carotene (mg/100 g)	$13.61 \pm 0.00$ <sup>a</sup>	$54.00\pm0.00~^{\rm b}$	$54.32\pm0.00~^{b}$	$54.42\pm0.01~^{\rm b}$

Table 3. The content of selected carotenoids in fresh and dried quince.

Values are means  $\pm$  standard deviation (n = 3). <sup>a–d</sup>—different letters in the same line are significantly different (Duncan's test, *p* < 0.05). Fresh—fresh fruit; Conv-50—convection-dried fruit at 50 °C; Conv-70—convection-dried fruit at 70 °C; Freeze-Dried—freeze-dried fruit.

Table 4. The content of selected flavonoids in fresh and dried quinces.

Flavonoids	Fresh	Conv-50	Conv-70	Freeze-Dried
Catechin (mg/100 g)	$3.33 \pm 0.09$ <sup>b</sup>	$3.10\pm0.03~^{\text{ab}}$	$2.95\pm0.02~^{a}$	$13.39\pm0.28~^{\rm c}$
Epigallocatechin (mg/100 g)	$8.04\pm0.05~^{\rm a}$	$14.96\pm0.61~^{\rm c}$	$10.89 \pm 0.23$ <sup>b</sup>	$30.69 \pm 0.62$ <sup>d</sup>
Rutoside-3- <i>O</i> - quercetin (mg/100 g)	$4.60\pm0.26$ a	$19.11 \pm 1.04$ <sup>d</sup>	$15.98\pm0.39^{\text{ b}}$	$17.34\pm0.25~^{\rm c}$
Quercetin (mg/100 g)	$0.95\pm0.01~^{\rm a}$	$2.74\pm0.02~^{\rm c}$	$2.34\pm0.06~^{b}$	$3.87\pm0.01~^{\rm d}$

Values are means  $\pm$  standard deviation (n = 3). <sup>a–d</sup>—different letters in the same line are significantly different (Duncan's test, *p* < 0.05). Fresh—fresh fruit; Conv-50—convection-dried fruit at 50 °C; Conv-70—convection-dried fruit at 70 °C; Freeze-Dried—freeze-dried fruit.

	<b>F</b> 1	<b>a</b>		E D! 1
Phenolic Acids	Fresh	Conv-50	Conv-70	Freeze-Dried
Gallic acid (mg/100 g)	$1.10\pm0.02~^{\text{a}}$	$5.17\pm0.15~^{\rm b}$	$4.68\pm0.05~^{\rm b}$	$17.68\pm0.17~^{\rm c}$
Chlorogenic acid (mg/100 g)	$14.47\pm0.14~^{\rm a}$	$45.34\pm1.41~^{\rm c}$	$39.20 \pm 0.38$ <sup>b</sup>	$244.12 \pm 7.85$ <sup>d</sup>
Caffeic acid (mg/100 g)	$0.61\pm0.00$ $^{\rm a}$	$3.79\pm0.10^{\text{ b}}$	$3.50\pm0.06~^{b}$	$9.83\pm0.15$ c $$
<i>p</i> -coumaric (mg/100 g)	$0.39\pm0.00~^{a}$	$6.73\pm0.08~^{\rm bc}$	$6.22\pm0.37~^{\rm b}$	$7.19\pm0.36$ c $^{\rm c}$
Ferulic acid (mg/100 g)	$0.31\pm0.00~^{a}$	$1.92\pm0.05~^{\rm b}$	$1.81\pm0.12~^{\rm b}$	$4.85\pm0.07~^{\rm c}$

Table 5. The content of selected phenolic acids in fresh and dried quince fruit.

Values are means  $\pm$  standard deviation (n = 3). <sup>a–d</sup>—different letters in the same line are significantly different (Duncan's test, *p* < 0.05). Fresh—fresh fruit; Conv-50—convection-dried fruit at 50 °C; Conv-70—convection-dried fruit at 70 °C; Freeze-Dried—freeze-dried fruit.

**Table 6.** The content of tannins, total polyphenols and antioxidant activity in fresh and dried quince fruit.

Bioactive Compounds	Fresh	Conv-50	Conv-70	Freeze-Dried
Tannins (g/100 g)	$3.64\pm0.06~^{a}$	$5.08\pm0.04~^{\rm b}$	$6.85\pm0.61~^{\rm c}$	$9.74\pm0.05~^{\rm d}$
Total polyphenols (mg GAE/100 g)	$364.53 \pm 3.76$ <sup>c</sup>	$220.15\pm2.30~^{\text{a}}$	$287.87 \pm 7.11$ <sup>b</sup>	$976.16 \pm 11.48$ <sup>d</sup>
Antioxidant activity (μM TEAC/100 g)	$520.78 \pm 8.56$ <sup>c</sup>	$426.08\pm9.92~^{\text{a}}$	$467.94 \pm 2.18$ <sup>b</sup>	$1478.08 \pm 6.24$ <sup>d</sup>

Values are means  $\pm$  standard deviation (n = 3). <sup>a–d</sup>—different letters in the same line are significantly different (Duncan's test, *p* < 0.05). Fresh—fresh fruit; Conv-50—convection-dried fruit at 50 °C; Conv-70—convection-dried fruit at 70 °C; Freeze-Dried—freeze-dried fruit; GAE—gallic acid equivalent; TEAC—Trolox-equivalent antioxidant capacity.

The quince was characterized by a high content of carotenoids (Table 3): the average sum of identified carotenoids in fresh fruit was  $89.96 \pm 0.37 \text{ mg}/100 \text{ g}$ , while in dried fruit it was  $127.27 \pm 52.42 \text{ mg}/100 \text{ g}$ , with the dominant carotenoids in all the tested samples being  $\beta$ -carotene, chlorophyll a and chlorophyll b. The research showed a significant (p < 0.05) effect of the drying method on the content of these components. The highest content of carotenoids was found in freeze-dried quinces ( $180.68 \pm 0.63 \text{ mg}/100 \text{ g}$ ), lower in Conv-50 ( $145.55 \pm 1.60 \text{ mg}/100 \text{ g}$ ) and the lowest in Conv-70 ( $137.88 \pm 1.02 \text{ mg}/100 \text{ g}$ ). The applied drying methods had a significant effect on the content of chlorophyll a, chlorophyll b and lutein (Table 3). The highest concentration of these components was found in freeze-dried fruit, while convection-dried fruit showed significantly (p < 0.01) lower content, on average by 35.2, 35.2 and 31.8%, respectively, with the lowest values for Conv-70 quinces. Despite the high sensitivity to the temperature, such relationships were not found in the case of  $\beta$ -carotene and zeaxanthin, the concentrations of which were at a very similar level in dried fruit, regardless of the drying method (on average  $54.37 \pm 0.04 \text{ mg}/100 \text{ g}$ ).

In the literature, no information was found on the general content of carotenoids or their profile in dried quinces, and there is little research on these components in fresh fruit. The total amount of carotenoids in the quince skin was 0.86–0.16 mg/100 g, while in the flesh it was 0.42–0.04 mg/100 g, which differed from the results obtained in our study, but the authors expressed the carotenoids content as carotene [27]. The study by Ponder and Hallmann (2017) [7] showed that in fresh quince the dominant carotenoid

was chlorophyll a  $(1.90 \pm 0.05 \text{ mg}/100 \text{ g f.m.} (f.m.—fresh matter))$ , followed by chlorophyll b  $(1.76 \pm 0.05 \text{ mg}/100 \text{ g f.w.})$ , which was confirmed in the research carried out in this work. The separation of carotenoids in the study of the same authors also showed  $1.46 \pm 0.07 \text{ mg}/100 \text{ g}$  of zeaxanthin,  $1.34 \pm 0.05 \text{ mg}/100 \text{ g}$  of  $\beta$ -carotene and the lowest content for lutein  $(0.62 \pm 0.06 \text{ mg}/100 \text{ g})$  [7], which was confirmed in these studies. Against the background of modest literature research, the results of carotenoid content and profile obtained in this study were higher, which could be determined by many factors, such as the variety, method and conditions of cultivation or harvesting, storage conditions and time, or the degree of fruit ripeness or the type of applied analytical procedure [7,27].

This study also examined the effect of various drying methods on the selected flavonoids content in quinces, and the obtained results are presented in Table 4.

The average content of flavonoids determined in fresh fruit was  $16.92 \pm 0.13 \text{ mg}/100 \text{ g}$ , while in dried it was  $45.79 \pm 15.04 \text{ mg}/100 \text{ g}$ , with significant (p < 0.05) differences between both the content and the profile of selected flavonoids in fresh and dried quinces. In dried fruit, the highest total amount of flavonoids was determined in the freeze-dried quinces ( $65.29 \pm 0.90 \text{ mg}/100 \text{ g}$ ), and it was almost two times lower in the convection-dried at 50 °C ( $39.91 \pm 1.67 \text{ mg}/100 \text{ g}$ ) and 70 °C ( $32.17 \pm 0.67 \text{ mg}/100 \text{ g}$ ).

As a result of the chromatographic separation of flavonoid compounds, epigallocatechin and rutinoside-3-O-quercetin were the dominant flavonoids in fresh and dried by various methods fruit; the content of catechin was about two times lower, and quercetin had the lowest share.

In the case of dried fruit, the highest content of epigallocatechin was found in freezedried fruit, and it was two times lower in Conv-50 and almost three times lower in Conv-70, which could suggest a significant effect of the drying method on the content of this bioactive ingredient. In the case of rutinoside-3-*O*-quercetin in dried fruit, a slightly different tendency was found than in the case of epigallocatechin, because the highest content of this component was found in Conv-50, and it was lower in freeze-dried samples and the lowest in Conv-70.

The highest (p < 0.05) amount of catechin was found in freeze-dried quinces, and it was more than four times lower in convectively dried quinces, although in this case the effect of drying temperature on the content of catechin was not detected (average  $3.03 \pm 0.08 \text{ mg}/100 \text{ g}$ ). The content of quercetin in the analyzed quince samples was characterized by the lowest variability, and again, its highest content was found in freeze-dried quinces and significantly (p < 0.05) lower in convective-dried ones.

The results obtained in this research were similar to those of other authors. The research by Ponder and Hallmann (2017) [7] showed  $34.69 \pm 1.95 \text{ mg}/100 \text{ g}$  of total flavonoids, and the authors also determined the content of selected compounds, obtaining values similar to the results in this study for rutinoside-3-*O*-quercetin and quercetin (respectively,  $8.57 \pm 0.54 \text{ mg}/100 \text{ g}$  and  $5.86 \pm 0.50 \text{ mg}/100 \text{ g}$ ). Moreover, in the studies by Wojdyło (2013) [3], the content of rutinoside-3-*O*-quercetin was similar to the values obtained in this work and amounted to 5.10 mg/100 g for the pulp and 20.28 mg/100 g for its fruit skin. The catechin content, in turn, ranged from 1.09 mg/100 g in fruit pulp to 37.85 mg/100 g in fruit skin. Similar values for the peel of quince were also obtained by Essafi-Benkhadir et al. (2012) [32], where the highest content of separated flavonoids was found for rutinoside-3-*O*-quercetin (47.21 ± 4.56 mg/100 g), and it was significantly lower for quercetin (7.01 ± 2.92 mg/100 g) or catechins ( $5.07 \pm 2.15 \text{ mg}/100 \text{ g}$ ). However, in the available literature no information was found on the effect of various drying methods on the content of flavonoids and their profile in quince.

Another of the analyzed groups of phenolic bioactive compounds in this work were phenolic acids. As shown in Table 5, the highest (p < 0.05) sum of determined phenolic acids was found in freeze-dried quinces (283.22 ± 7.56 mg/100 g), and it was lower in dried by convection, i.e., more than four times (Conv-50) and five times (Conv-70) lower than in the freeze-dried samples (63.41 ± 1.53 mg/100 g and 55.40 ± 0.18 mg/100 g, respectively), which allowed us to conclude that the drying conditions played a significant

role in preserving these bioactive ingredients in dried fruit. Although no information on changes in the content of phenolic acids in quinces due to dehydration methods was found in the available literature, studies by other authors have shown that various drying methods

significantly affect the quality and content of bioactive ingredients in other fruit [73,74]. As a result of the chromatographic separation of phenolic acids (Table 5), the tested samples of quince were characterized by a varied profile of phenolic acids, in which chlorogenic acid was dominant. The remaining acids were present in much lower concentrations, and the smallest share in all analyzed samples of quince (fresh, dried) was ferulic acid. The trends obtained in this research were confirmed in the studies of other authors, who also showed the highest amount of chlorogenic acid and its derivative, i.e., neochlorogenic acid, in the profile of phenolic acids in fresh quinces [3].

This study showed the highest content of these components in freeze-dried fruit, which were characterized by the highest (p < 0.05) amount of chlorogenic, gallic, p-coumaric, coffee and ferulic acids. Convective drying led to a significant (p < 0.05) decrease in the content of all determined phenolic acids, but no significant differences were found between convective drying at 50 °C and 70 °C. Thus, the content of chlorogenic acid decreased almost six times (average 42.27 ± 3.49 mg/100 g), gallic acid almost four times (average  $4.92 \pm 0.29$  mg/100 g) and almost three times in the case of caffeic (average  $3.65 \pm 0.17$  mg/100 g) and ferulic (average  $1.86 \pm 0.10$  mg/100 g) acids in comparison with freeze-dried fruit. The smallest differences were found for p-coumaric acid, the content of which in convection-dried fruit was lower by about 10% compared to freeze-dried fruit.

The available literature lacks data on the profile of phenolic acids in dried quinces depending on the various drying methods, and there are also scarce data on these phenolic compounds in quince in general. In the research conducted by Ponder and Hallmann (2017) [7], the total content of phenolic acids in quinces was  $34.69 \pm 1.95 \text{ mg}/100 \text{ g}$ , which was almost twice as high as in the fresh fruit tested in this study ( $16.88 \pm 0.16 \text{ mg}/100 \text{ g}$ ). These authors also showed the content of gallic, chlorogenic, caffeic and *p*-coumaric acids at the levels of  $3.27 \pm 0.24 \text{ mg}/100 \text{ g}$ ,  $3.84 \pm 0.33 \text{ mg}/100 \text{ g}$ ,  $6.82 \pm 1.84 \text{ mg}/100 \text{ g}$  and  $20.75 \pm 2.66 \text{ mg}/100 \text{ g}$ , respectively, which in the analysis of the entire profile differed from the results obtained in our study.

In the studies of Ponder and Hallmann (2017) [7], the highest content was found in *p*-coumaric acid, while in our study it was present in one of the lowest concentrations in fresh fruit (0.39  $\pm$  0.00 mg/100 g). According to the literature, differences in both the content of bioactive compounds and their proportions may result from many factors, e.g., genetic and environmental factors, as well as be strongly correlated with the degree of fruit ripeness [3,39,77]. In contrast, in the studies of Wojdyło et al. (2013) [3], the content of chlorogenic acid in the flesh of quince fruit was 183.79 mg/100 g, and more than twice as high in the skin of the fruit (314.46 mg/100 g). However, in the literature, the content of this ingredient is also within very wide limits, for example, in the study of Essafi-Benkhadir et al. (2012) [32], it was 12.85  $\pm$  0.16 mg/100 g, which was similar to the results of our own research.

The content of total polyphenolic compounds and antioxidant activity (by spectrophotometry) as well as the content of tannins in fresh quince and in dried quince were also examined in this study, and the obtained results are presented in Table 6.

Based on the obtained results, it can be concluded that fresh quince was characterized by a high content of total polyphenols, but the results obtained in this study differed from the data available in the literature. Al-Zughbi and Krayem (2022) [17] showed a lower content of total polyphenols in quince pulp (68.14 mg GAE/100 g), which could be due to the fact that in our research fresh quince also contained a peel, and higher (according to the literature) content of polyphenolic compounds compared to fruit pulp [32,33]. In the research of Stojanović et al. (2017) [39], the content of total polyphenols in various cultivars of common quince, both in their pulp and in the skin of the quince, was in the ranges of 71.03–158.89 mg GAE/100 g and 140.12–202.92 mg GAE/100 g, respectively. The total content of polyphenols in the study by Szychowski et al. (2014) [77] in the pulp of

quince was 44.8–101 mg GAE/100 g, depending on the variety of quince. In quince skin, these values were much higher and ranged from 327 to 581 mg GAE/100 g. Stojanović et al. (2017) [39] obtained a similar content of total polyphenols in the fruit pulp but showed a much higher content of these components in the peel than Szychowski et al. (2014) [77].

In the studies of other authors, the total polyphenol content in fresh quinces was around 350 mg GAE/100 g, which in turn was a value similar to that obtained in our research for fresh fruit [3,27,30,33]. Differences in the content of bioactive ingredients, including polyphenols, are determined by both genetic (species, variety, genotype) and environmental factors (climate, growing or harvesting conditions) [10,27,30,77]. Maturity is also key. The research by Wojdyło (2013) [3] showed significant changes in the content of both phenolic acids and flavonols, and thus also total polyphenols, depending on the degree of ripeness of quince fruit.

The available literature lacks data on the effect of various drying methods on the total polyphenol content or the antioxidant potential of quinces. As a result of the conducted research (Table 6), the highest content of total polyphenols was found in freeze-dried quince, and convectively dried fruit had significantly (p < 0.05) lower amounts of these components (3.4 times and 4.4 times lower, respectively, for Conv -70 and Conv-50). The tested quince samples were also characterized by high antioxidant activity, ranging from  $426.08 \pm 9.92$  to  $1478.08 \pm 6.24 \,\mu\text{M}$  TEAC/100 g, with the highest antioxidant potential in freeze-dried quinces. The antioxidant potential of both fresh and dried quinces was significantly (p < 0.05) correlated with the content of total polyphenols and showed similar tendencies of changes for this parameter. On the basis of the conducted results, the correlation between the content of total polyphenols (mg GAE/100 g) and the antioxidant activity measured by the ability to deactivate synthetic ABTS<sup>+•</sup> radicals (µM TEAC/100 g) was calculated, obtaining a high correlation coefficient R<sup>2</sup> for this relationship, amounting to 0.9636. This relationship is well known and described in the literature on the content of polyphenols and their antioxidant activity in various raw materials and products of plant origin [77,83,84], also in quinces [3,17].

Fresh quinces are relatively rarely eaten in an unprocessed form, usually due to their high hardness or bitterness, but mainly due to their astringent taste [77], given to them by various phytochemicals, including tannins [85]. Table 6 shows the content of tannins in the tested samples of quince, fresh fruit and fruit dried by various methods.

According to the conducted research, fresh fruit was characterized by a high tannin content of  $3.64 \pm 0.06$  g/100 g. The highest (p < 0.05) amount of these components was found in freeze-dried fruit, containing  $9.74 \pm 0.05$  g/100 g of tannins. The high concentration of these ingredients in freeze-dried fruit was most likely caused by, among other things, high dehydration and dry matter concentration ( $98.58 \pm 0.18\%$ ), the highest among all tested samples of processed quince (Table 1). In the available literature, no information was found on the content of tannins in dried quince. There are also only scant data on the content of tannins in general. In addition, their comparison with the results obtained in the conducted research is very difficult due to the use of various analytical methods to determine their quantity. Nevertheless, the content of tannins in the studies of Djilali et al. (2021) [85] ranged from 7.33% to 9.7%, and therefore was similar to those obtained in our study, but the authors determined them with two different methods (precipitation and colorimetric) expressing the results as the difference between the total polyphenol content and the total polyphenol content after tannin complexation with casein.

The available literature does not contain any data on the effect of various drying methods of quinces or other fruit materials on the content of these phytochemicals. In the conducted research, the amount of tannins in convection-dried quinces was significantly (p < 0.05) lower than in freeze-dried quinces, and the content of these components was almost 30% lower in Conv-70 and almost 48% lower in Conv-50 compared to samples of freeze-dried quinces.

According to the literature, tannins occur in plants in two main forms, i.e., hydrolyzing, which includes gallotannins and ellagitannins, i.e., derivatives of gallic and ellagic acids,

respectively, esterified with sugars (mainly glucose) and non-hydrolyzing (condensed), called proanthocyanidins, which include various condensation products, e.g., catechins, derivatives of flavan-3-ol, with varying degrees of polymerization [86]. These substances are a component of cell walls and are very common in the world of plants, occurring in the bark of trees, skin and the vegetative organs of plants, as well as in fruits, and they protect plants against bacterial and fungal infections and also scare away herbivores due to the formation of acidic, bitter and astringent complexes with salivary proteins [87]. According to the available literature, both their content and profile (hydrolyzing/non-hydrolyzing) change in fruits during their ripening; they can also be leached from plants and thus their level can be reduced [86].

In the literature, e.g., the bactericidal, fungicidal and insecticidal effect of tannins contained in quinces and weak effect of extracts containing tannins on *Escherichia coli*, *Staphylococcus aureus* and *Candida albicans* strains is demonstrated. However, an increase in the mortality rate of *Tribuliumm confusum* (a food pest that feeds on cereals, flour, bran, dry bread, attacking mills, warehouses or granaries) was observed along with the increase in the concentration of quince tannins in the solution [85]. The authors showed that extracts containing tannins from common quinces affected the integrity of the digestive tract in the tested insects, causing malformations in offspring and the death of adults, thus demonstrating their effective insecticidal activity against *Tribulium confusum*. These studies suggest potential opportunities to use quince tannins as an alternative to chemical insecticides without the need for pesticides to control biological pests [85].

According to the literature, tannins, due to their astringent properties, are used in the external treatment of inflammation and skin injuries, while their consumption can prevent various chronic diseases. By delaying the absorption of glucose in the small intestine, they lower blood sugar levels, thanks to which they can act as an antidiabetic, entering into direct and indirect reactions with the fat tank, and tannins contained in the diet can also protect against obesity [87]. Tannins are compounds that can act in the digestive tract, showing antioxidant, free radical scavenging and antibacterial properties [84,86,87]. Dietary supplements containing plant extracts and tannins are available on the market, examples of which are supplements containing pomegranate seed extract, recommended in the prevention and reduction of atherosclerosis, green coffee extract supporting weight loss or cranberry extract inhibiting the development of urinary tract infections [85]. Taking into account the above considerations regarding the pro-health effect of tannins and the obtained research results, freeze-dried quinces with a concentrated content of tannins could be used as a valuable ingredient and additive to functional food.

#### 4. Conclusions

The results obtained in this study provide valuable information on the physicochemical and bioactive properties of fresh fruit and those subjected to drying using various methods. Quince fruit, due to their health-promoting properties, resulting primarily from their high density of bioactive components, such as carotenoids, tannins and polyphenolic compounds, with strong antioxidant properties, can be a valuable component of functional foods or dietary supplements. Incorporating quince fruit in their traditional form or their processing products in dried form into the daily diet can be an important element in the prevention of many civilization diseases. Due to their specific physicochemical and sensory properties, including above all the hardness of their flesh, their bitterness and their astringency, quince fruit are rarely consumed fresh, and far more often in the form of various products and preparations, such as marmalades, jam, jams and jellies, or additives to cakes, desserts or beverages.

**Supplementary Materials:** The following supporting information can be downloaded at: https: //www.mdpi.com/article/10.3390/agriculture13020446/s1, Figure S1: The picture of spectra of polyphenols analysis in fresh quince fruit; Figure S2: The picture of spectra of polyphenols analysis in quince fruit dried in 50 °C; Figure S3: The picture of spectra of carotenoids analysis in quince fruit dried in 70 °C; Figure S4: The picture of spectra of carotenoids analysis in quince fruit freeze-dried; Figure S5: The picture of spectra of carotenoids analysis in fresh quince fruit; Figure S6: The picture of spectra of polyphenols analysis in quince fruit dried in 50 °C; Figure S7: The picture of spectra of carotenoids analysis in quince fruit dried in 70 °C; Figure S8: The picture of spectra of carotenoids analysis in quince fruit freeze-dried.

**Author Contributions:** Conceptualization, K.N. and S.A.; methodology, K.N., E.H., A.S. (Anna Sadowska) and A.S. (Arkadiusz Szterk); formal analysis, K.N., E.H., A.S. (Anna Sadowska), A.S. (Arkadiusz Szterk), K.B. and S.A.; resources, K.N.; data curation, K.N.; writing—original draft preparation, K.N., A.S. (Anna Sadowska), A.S. (Arkadiusz Szterk), K.B. and B.W.-R.; writing—review and editing, K.N., A.S. (Anna Sadowska), A.S. (Arkadiusz Szterk), K.B. and B.W.-R.; visualization, K.N., A.S. (Anna Sadowska), A.S. (Arkadiusz Szterk), K.B. and B.W.-R.; visualization, K.N., A.S. (Anna Sadowska), A.S. (Arkadiusz Szterk), K.B. and B.W.-R.; visualization, K.N., A.S. (Anna Sadowska), A.S. (Arkadiusz Szterk) and K.B. All authors have read and agreed to the published version of the manuscript.

**Funding:** This research was prepared as part of the statutory activity of the Department of Functional and Ecological Food, Warsaw University of Life Sciences.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: Not applicable.

Conflicts of Interest: The authors declare no conflict of interest.

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