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Impact of Incorporating Two Types of Dried Raspberry Pomace into Gluten-Free Bread on Its Nutritional and Antioxidant Characteristics

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Abstract: The purpose of this study was to determine the impact of the pomace drying methods (freeze-drying and convection-drying) and their percentage (0–10%) on selected physicochemical properties of gluten-free bread. The contents of nutrients (protein, fiber, fat, ash, and carbohydrates), bioactive compounds, antioxidant properties, acidity, baking efficiency, and moisture of the obtained products were determined. Fortifying the bread with raspberry pomace resulted in a change in fiber content from 18.13% d. b. (control sample) up to 19.97% d. b. (10% of freeze-dried pomace), and a change in the fat and ash content in the bread from 5.74% and 2.83% d. b. (control sample) to 7.18% and 3.12% d. b. (10% of freeze-dried pomace). The content of carbohydrates decreased after adding raspberry pomace to the bread, from 65.71% d. b. (control sample) to 63.68% d. b. (5% of freeze-dried pomace). The research carried out also showed that the introduction of 10% freeze-dried raspberry pomace increased the total polyphenol content by 81.75% and the antioxidant properties defined by the ABTS method by 159.54% and by the DPPH method by 96.43% compared to the control bread. The introduction of pomace resulted in a significant reduction in the total baking loss, from 15.1% to 10.62%, and an increase in the total titratable acidity of the crumb, from 2.13 mL $_{NaOH}$/10 g d. b to 7.78 mL $_{NaOH}$/10 g d. b. Principal component analysis highlighted a marked effect of the drying method and content of raspberry pomace on the quality values of gluten-free bread. This research demonstrated that raspberry pomace can be a valuable source of fiber and bioactive substances in gluten-free bread.

Keywords: raspberry by-products; gluten-free bread; antioxidant activity; polyphenols; fat; fiber; protein; nutritional properties; bioactive compounds; principal component analysis (PCA)

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1. Introduction

In recent years, the popularity of fruit preserves has been growing, including juices, purees, and smoothies, resulting in an increase in production waste, which is pomace. Fruit and vegetable processing produces significant amounts of by-products, which represent approximately 25% to 30% of the entire commodity group [1]. The disposal of unused parts of the raw material is a significant problem for the fruit industry. In order to ensure a closed system and reduce food waste, by-products, such as fruit pomace, from the food industry can be re-used [2]. Food by-products are a rich source of antioxidant compounds and nutrients, including fiber, and can, therefore, be used as alternative ingredients to develop innovative recipes with an increased, positive impact on health [3–7]. Numerous studies indicate the use of functional ingredients obtained from fruit pomaces to improve the nutritional profile of food products [8–11]. By using pomace in the food industry, it is possible to reduce manufacturing expenses and create an alternative food source [12].

Fruit pomace is a valuable by-product with great potential, but it contains significant amounts of water, which makes it unstable [13–19]. The methods used to preserve by-products can significantly extend their durability, increase the possibility of re-use, and influence the properties and composition of pomace. Drying is considered one of the traditional but also most effective techniques for perishable fruits. Drying strongly inhibits the activity of microorganisms and enzymes, prolongs the shelf life, facilitates the management and distribution of by-products, and allows obtaining many functional ingredients as added value for use in the food sector [20]. Research carried out so far shows that the freeze-drying method has a positive impact on the content of nutrients in pomace and finished products [21,22].

For several years, research has been conducted on the possibility of using fruit pomace in bread production due to the high content of anthocyanins, flavonoids, and ascorbic acid. Fruits are added in fresh, dried, powder, flour, or extract form [23]. The vast majority of literature reports concern the enrichment of traditional white bread with fruits, such as pear [24], apricot kernels [25], pomelo [11], baobab fruit, grape seed [26], banana [27,28], sea-buckthorn, elderberry, hawthorn, rowan [29], blueberry [30], garcinia cowa fruits [31], blackcurrant [32], apple pectin, kiwifruit [33], and chokeberry [10]. The impact of fruit pomace on the properties of gluten-free bread is much less researched. However, it should be noted that fruit industry by-products are gluten-free, which makes them potentially ideal suitable components for developing innovative food for patients with celiac disease [34–37]. O'Shea et al. [38] analyzed flour from orange pomace, Gumul et al. [39] and Cantero et al. [40] added apple pomace, Djeghim et al. [41] also dealt with apple and orange pomace, and Korus et al. [42] with blackcurrant and strawberry pomace.

Nowadays, consumers have an increasing sense of responsibility for the state of the natural environment and, therefore, for sustainable development. The increase in their nutritional awareness is influenced primarily by the search and selection of products that meet the requirements of a healthy and balanced diet [43,44]. Food products that provide additional health benefits are desirable, which often forces changes to the recipes or technology of traditional products, which undoubtedly include bakery products. Modification of traditional recipes by enhancing healthy components has a positive impact on obtaining a product with functional features that is suitable for consumption [45]. Fortifying baked goods with natural antioxidants provides positive effects on health. As a consequence, there is a rise in the intake of bioactive substances from bakery products that traditionally lack these compounds, leading to enhanced consumption by consumers. Such enrichment of bakery products may affect not only the physicochemical properties but the functional characteristics and acceptability by consumers as well [46–48].

Red raspberries (*Rubus idaeus* L.) are berries that contain many nutrients and bioactive ingredients. At the same time, they are one of the largest sources of dietary fiber, the content of which is 6.5 g/100 g of fresh weight (12.5 g/100 kcal). Additionally, they possess nutrients such as vitamin C, magnesium, potassium, vitamin K, calcium, and iron. Red raspberries have a unique polyphenol profile, which is marked by the content of anthocyanins and ellagitannins. Red raspberry fruits, which are a source of diverse extracts and unique ingredients, have anti-inflammatory, antioxidant, and metabolism-stabilizing effects [49–53]. In Europe, Poland holds the position of being the second-largest raspberry producer. The vast majority of fruit is industrially processed (78%), of which juice production accounts for up to 27% [54]. Such a huge amount of waste not only generates high costs but is also an environmental problem.

The concept of a closed-loop bioeconomy appears to offer a favorable resolution to the issue of food waste. This approach is centered around the principles of reduction, recycling, and re-use [55]. The search for new ways of using raspberry pomace is a current subject of scientific research.

The available literature reports did not compare the influence of the pomace drying methods on the properties of bread. There is also no information about the possibility of using raspberry pomace in gluten-free bread. Therefore, the purpose of this study is to assess the impact of the addition of convective- and freeze-dried raspberry pomace on the nutritional and antioxidant properties of gluten-free bread.

2. Materials and Methods

2.1. Raw Materials

The plant raw material consisted of 'Polesie' raspberry fruits, which were purchased in August 2023 from one of the organic farms in the Lublin Voivodeship (Poland). Immediately after harvesting, the raspberries were pressed on a basket press (810509 Browin, Łódź, Poland). Part of the obtained pomace was subjected to convectional drying using the Pol-Eko Aparatura SLW 115 Top + (Wodzisław Śląski, Poland; 48 h, 30 °C) and the second part was subjected to freeze-drying. Before freeze-drying, the raspberry pomace was frozen to a temperature of −40 °C, as plates about 1 cm-thick, using a Memmert CTC256 climatic chamber (Schwabach, Germany). Then, the samples were freeze-dried in a Martin ChristAlpha 2-4 LD plus device (Osterode am Harz, Germany) at a pressure of 20 Pa for 72 h. In order to preserve as many thermolabile bioactive compounds as possible during drying, the shelves were not heated. A laboratory grinder (Chemland, FW100, Stargard, Poland) was used to grind the pomace.

2.2. Preparation of Dough and Bread Baking Procedure

The bread was made and labeled according to the information provided in Table 1. The bread dough was made in a laboratory spiral mixer (Kenwood, Havant, UK) and the components were mixed for 5 min. The dough was divided into 1075 g portions, which were stored in a mold for post-fermentation (time: 40 min, temperature: 37 °C, and relative humidity: 80%). Subsequently, the breads were baked in a convection-steam oven (Houno, Randers, Denmark) at a temperature of 230 °C for a duration of 40 min.

Table 1. Model of experiment parameters.

Probe Code	0	5CD	10CD	5FD	10FD
Ingredients	**Amount of Ingredients**				
Rice flour	250 g	225 g	200 g	225 g	200 g
Corn flour	200 g	200 g	200 g	200 g	200 g
Potato starch	50 g	50 g	50 g	50 g	50 g
Water	500 g	500 g	500 g	500 g	500 g
Rapeseed oil	30 g	30 g	30 g	30 g	30 g
Dry yest	8 g	8 g	8 g	8 g	8 g
Salt	12 g	12 g	12 g	12 g	12 g
Sugar	10 g	10 g	10 g	10 g	10 g
Ground flax seeds	15 g	15 g	15 g	15 g	15 g
Convection-dried raspberry pomace	0 g	25 g	50 g	0 g	0 g
Freeze-dried raspberry pomace	0 g	0 g	0 g	25 g	50 g

0—control bread; 5CD—bread with 25 g of convection-dried raspberry pomace added; 10CD—bread with 50 g of convection-dried raspberry pomace added; 5FD—bread with 25 g of freeze-dried raspberry pomace added; 10FD—bread with 50 g of freeze-dried raspberry pomace added.

2.3. Determination of Physical Properties of Bread

2.3.1. Determination of Bread Moisture

The weighing method was used to assess the moisture content of bread. For this purpose, material samples weighing 2 g were dried using a laboratory dryer (Pol-Eko, SLN 15, STD, Wodzisław Śląski, Poland) at a temperature of 130 °C until a weight loss was noticed [56,57].

2.3.2. Determination of Total Baking Loss

The total baking loss, X (%), was determined according to the equation:

$$X = \frac{(a - b)\cdot 100}{a}$$

where:
a—mass of dough formed for baking,
b—mass of cooled bread [58].

2.3.3. Determination of Titratable Acidity (TTA)

Titratable acidity (TTA) was measured in 10 g of bread by extraction with distilled water and titrated with 0.01 N NaOH, raising the pH to 8.5. TTA was expressed as mL $_{NaOH}$/10 g [59].

2.4. Determination of Nutritional Properties of Bread

2.4.1. Determination of Dietary Fiber Content

The determination of the fiber content was carried out according to the weighing method following the PN-A-79011-15:1998 standard. The analysis consisted of digesting the test sample with enzymes, such as thermostable α-amylase, pepsin, and pancreatin, and then determining the undigested residue of insoluble dietary fiber and soluble dietary fiber by weight after precipitation from the supernatant solution [56].

2.4.2. Protein Content Analysis

Protein content was assessed using the Kjeltec analyzer (TM8400) and ASN 3100 software. Distillation was performed in the automatic Kjeltec Auto Set by Tecator. The nitrogen content was converted to protein with the conversion rate N $\times$ 6.25 [57].

2.4.3. Determination of Fat Content

The Soxtec apparatus (TM8000) was used to determine the total fat content. The study was carried out using the continuous ether extraction method. AN 310 software was used to analyze the results.

2.4.4. Ash Content Analysis

The determination of the ash content consisted of complete combustion of the material and roasting the ash in a muffle furnace (LAC Ltd., M: LE 18/11).

2.4.5. Determining the Carbohydrate Content

The proximate carbohydrate content (C) was assessed from the following difference: 100 − (weight in grams (protein + fat + TDF + ash) in 100 g of dry matter).

2.4.6. Calculation of Energy Value

The energy value (EV) was fixed on the grounds of macronutrient content, using the equation: energy value (kcal) = 4 $\times$ (gprotein + gcarbohydrates) + 9 $\times$ (gfat) + 2 $\times$ (gTDF) [60].

The result was calculated and expressed in kcal/100 g of fresh weight of bread and in kcal/100 g of raspberry pomace after convection- or freeze-drying.

2.5. Determination of Antioxidant Properties of Bread

2.5.1. Preparation of Extracts for Chemical Analysis

The extracts were prepared by adding 2 g of bread/1 g of raspberry pomace to 30 mL of methanol and leaving them for 24 h. The next step was to pour the extract into another flask and add another 30 mL of methanol to the raffinate. Following a one hour extraction on a magnetic stirrer, the extract was gathered, combined with the previous extract portion, and subjected to centrifugation using a centrifuge (Neuation, iFuge D06, Gujarat, India) at 6500 rpm for 15 min.

2.5.2. Total Phenolic Content (TPC) Assay

The TPC value was assessed following the methodology proposed by Singleton et al. [61], with a modification made by Kobus et al. [62]. To determine the TPC of pomace, 0.2 mL of the extract was taken, and when determining the TPC of bread, 1.5 mL was taken. TPC was expressed as mg gallic acid equivalent per 1 g of dry matter (mg GAE g^{-1} dry matter).

2.5.3. Antioxidant Activity—ABTS and DPPH Assays

Antioxidant activity using the ABTS test was assessed according to the methodology described by Krzywicka and Kobus [63]. ABTS was expressed as a Trolox equivalent in µg per g of dry matter (µg TE/g d. m.).

Antioxidant activity using the DPPH test was assessed according to the methodology described by Krzywicka and Kobus [63]. To determine the DPPH of pomace, 60 µL of the extract was taken, and when determining the DPPH of bread, 400 µL was taken. DPPH was expressed as a Trolox equivalent in µg per g of dry matter (µg TE/g d. m.).

2.5.4. Total Anthocyanin Content (TAC) Assay

The total anthocyanin content was performed following the methodology described by Kobus et al. [62]. To determine the TAC of pomace, 0.3 mL of the extract was taken, and when determining the TAC of bread, 0.9 mL was taken. TAC was expressed as a cyanidin 3-glucoside equivalent in mg per g of dry matter (mg Cy3-GE/g d. m.).

2.6. Statistical Analyses

All tests were performed in triplicate. The obtained results were subjected to analysis of variance (ANOVA). The significance of the variations among the mean values was assessed using Tukey's test at a significance level of $p < 0.05$. Additionally, the principal component analysis (PCA) was carried out. All statistical analysis were performed with Statistica software (Statistica 13; StatSoft Inc., Tulsa, OK, USA).

3. Results and Discussion

Figure 1 shows a photo of baked gluten-free breads. The 5FD and 10FD breads had slightly different shapes. A change in the color of the bread fortified with by-products to a darker color was also observed. The porosity of the 10FD bread crumb in the visual assessment was lower compared to the other samples.

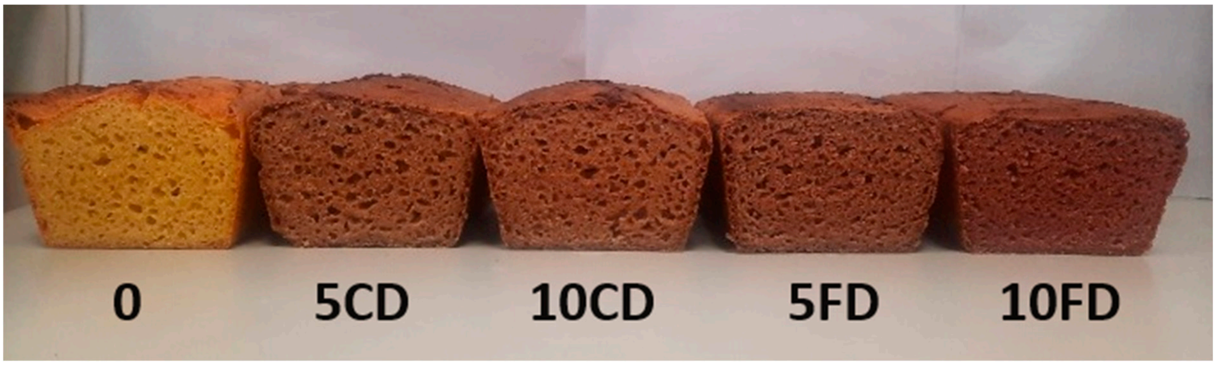

Figure 1. Cross-section appearance of breads with different raspberry by-product contents.

3.1. Physical Properties of Bread

The physical properties of the obtained gluten-free breads with the addition of raspberry pomace are shown in Table 2.

Table 2. Physical properties of breads.

Product	Probe	Crumb Moisture ($g \cdot 100\ g^{-1}$)	Total Baking Loss ($g_{loss} \cdot 100\ g^{-1}$)	TTA (mL $_{NaOH}$/10 g d. b.)
Bread	0	51.1 ± 0.64 [a]	15.1 ± 0.42 [a]	2.13 ± 0.18 [c]
	5FD	51.1 ± 0.25 [a]	11.7 ± 0.35 [b]	4.18 ± 0.11 [b]
	10FD	51.1 ± 0.35 [a]	10.6 ± 0.57 [b]	7.78 ± 0.11 [a]
	5CD	51.6 ± 0.13 [a]	11.4 ± 0.21 [b]	4.48 ± 0.18 [b]
	10CD	51.4 ± 0.24 [a]	11.5 ± 0.57 [b]	7.70 ± 0.14 [a]

0—control probe; 5FD—gluten-free bread with the addition of 5% freeze-dried raspberry pomace; 10FD—gluten-free bread with the addition of 10% freeze-dried raspberry pomace; 5CD—gluten-free bread with the addition of 5% convection-dried raspberry pomace; 10CD—gluten-free bread with the addition of 10% convection-dried raspberry pomace. The values of each parameter with different superscript letters in columns are significantly different (Tukey's test, $p \leq 0.05$).

The results of the ANOVA indicated no significant changes ($p = 0.344$) in the crumb moisture of gluten-free bread fortified with raspberry pomace. The average values of the tested features ranged from 51.1% (sample 5CD) to 51.6% (sample 5FD). In the study by Cantero et al. [40] regarding the use of 5% to 8% of apple pomace additives for gluten-free bread, the water content ranged from 48.15% with a 6% addition of pomace to 50.24% in bread with a 5% addition of pomace. These changes, similar to our experience, were not statistically significant. In turn, research on gluten-free breads with various additions of by-products, conducted by Djeghim et al. [41], showed that the moisture content of fortified bread decreased significantly in most tests. However, after using 5% and 7.5% tomato peel and 2.5% and 5% pepper peel, the average values of this feature did not change significantly compared to the control gluten-free bread.

The total weight loss of the tested gluten-free bread decreased after the addition of raspberry pomace. These changes were statistically significant. However, there was no significant impact of the pomace drying method or the percentage of raspberries added on changes in baking loss. Weight loss in the process of baking bread is usually related to water loss. The weight loss of bread can be reduced by adding by-products with a significant fiber content, which increases the ability to bind water during the baking process [64].

The introduction of 5% raspberry pomace to the gluten-free bread recipe resulted in an increase in the TTA of the crumb. Increasing the share of by-products to 10% resulted in a further significant increase in the TTA of gluten-free bread. However, there was no significant effect of the drying method on changes in the TTA of the gluten-free bread. A significant increase in the TTA of gluten-free bread fortified with raspberry pomace was caused by the high acidity of fruit residues. It was calculated that for freeze-dried raspberry pomace, the average TTA value was 12.5 mL $_{NaOH}$/1 g d. b., while for convection-dried raspberry pomace, it was 14.5 mL $_{NaOH}$/1 g d. b. Similar relationships were observed in the work of Majzoobi et al. [65], who analyzed the effect of adding carrot pomace on the properties of gluten-free dough. The study showed that the pH value of gluten-free cakes was reduced, which indicates an increase in the acidity of the material. The authors explained these changes by the presence of organic acids in plant residues. Additionally, in the experiment by Djeghim et al. [41], the addition of apple, orange, tomato, and pepper residues significantly reduced the pH value. The average pH values of gluten-free bread with 7.5% apple pomace were lower by 4.3% compared to the control sample.

3.2. Nutritional Properties of Bread and Pomace

The nutritional properties of the obtained gluten-free breads and raspberry pomace are shown in Tables 3 and 4.

Table 3. Content of dietary fiber in bread and pomace.

Product	Probe	IDF ($\%_{d.\ w.}$)	SDF ($\%_{d.\ w.}$)	TDF ($\%_{d.\ w.}$)
Bread	0	16.2 ± 0.22^{ab}	1.94 ± 0.03^{d}	18.1 ± 0.25^{b}
	5FD	15.9 ± 0.05^{b}	2.53 ± 0.03^{ab}	18.5 ± 0.09^{ab}
	10FD	17.2 ± 0.03^{a}	2.73 ± 0.06^{a}	20.0 ± 0.09^{a}
	5CD	15.6 ± 0.68^{b}	2.18 ± 0.15^{cd}	17.8 ± 0.83^{b}
	10CD	16.4 ± 0.05^{ab}	2.33 ± 0.06^{bc}	18.7 ± 0.09^{ab}
Pomace	FD	40.2 ± 0.11^{A}	6.17 ± 0.01^{A}	46.4 ± 0.04^{A}
	CD	39.3 ± 0.11^{B}	5.60 ± 0.02^{B}	44.9 ± 0.13^{B}

0—control probe; 5FD—gluten-free bread with the addition of 5% freeze-dried raspberry pomace; 10FD—gluten-free bread with the addition of 10% freeze-dried raspberry pomace; 5CD—gluten-free bread with the addition of 5% convection-dried raspberry pomace; 10CD—gluten-free bread with the addition of 10% convection-dried raspberry pomace. FD—freeze-dried raspberry pomace; CD—convection-dried raspberry pomace; IDF—insoluble dietary fiber; SDF—soluble dietary fiber; TDF—total dietary fiber. The values of each parameter with different superscript letters in columns are significantly different (Tukey's test, $p \leq 0.05$).

Table 4. Nutritional properties of bread and pomace.

Product	Probe	Protein ($\%_{d.\ w.}$)	Fat ($\%_{d.\ w.}$)	Ash ($\%_{d.\ w.}$)	C ($\%_{d.\ w.}$)	EV (kcal·100-1gf.w.)
Bread	0	8.43 ± 0.01^{a}	5.74 ± 0.06^{b}	2.83 ± 0.04^{a}	65.7 ± 0.15^{a}	171 ± 0.59^{a}
	5FD	8.40 ± 0.03^{a}	7.05 ± 0.26^{a}	2.98 ± 0.02^{b}	63.7 ± 0.14^{b}	173 ± 0.82^{a}
	10FD	8.51 ± 0.02^{a}	7.18 ± 0.02^{a}	3.12 ± 0.06^{c}	63.9 ± 0.04^{c}	171 ± 0.17^{a}
	5CD	8.44 ± 0.01^{a}	6.81 ± 0.01^{a}	2.95 ± 0.00^{ab}	64.8 ± 0.83^{ab}	173 ± 1.58^{a}
	10CD	8.48 ± 0.04^{a}	6.92 ± 0.06^{a}	3.02 ± 0.03^{bc}	63.9 ± 0.16^{b}	172 ± 0.01^{a}
Pomace	FD	9.73 ± 0.02^{A}	5.97 ± 0.05^{A}	2.39 ± 0.02^{A}	38.6 ± 0.11^{A}	235 ± 0.69^{A}
	CD	9.59 ± 0.08^{A}	5.99 ± 0.00^{A}	2.32 ± 0.02^{B}	40.3 ± 0.02^{B}	231 ± 0.15^{B}

0—control probe; 5FD—gluten-free bread with the addition of 5% freeze-dried raspberry pomace; 10FD—gluten-free bread with the addition of 10% freeze-dried raspberry pomace; 5CD—gluten-free bread with the addition of 5% convection-dried raspberry pomace; 10CD—gluten-free bread with the addition of 10% convection-dried raspberry pomace. FD—freeze-dried raspberry pomace; CD—convection-dried raspberry pomace; C—carbohydrate content; EV—energy value. The values of each parameter with different superscript letters in columns are significantly different (Tukey's test, $p \leq 0.05$).

There were no significant changes in the content of insoluble fiber in bread with raspberry pomace compared to the control sample. In turn, enriching the bread with raspberry residues significantly increased the amount of soluble fiber (SDF). This increase was statistically significant for both freeze-dried and convection-dried pomace. Moreover, a significantly higher share of SDF was found in the case of bread fortified with freeze-dried pomace compared to that with convection-dried pomace. Cantero et al. [40] indicated that the use of 8% apple pomace in gluten-free bread affected the fiber content, increasing its content from 3.65 g/100 g for the control bread to 8.15 g/100 g. Research results of Korus et al. [42] also indicated an increase in the total fiber content in gluten-free bread after the addition of fruit pomace. Fortification of bread with a 10% addition of blackcurrant pomace resulted in an increase in fiber content by 91.8%, and with a 10% addition of strawberry pomace, an increase in fiber content by 126.7%. Slightly smaller changes were observed in the experiment of O'Shea et al. [38], where the authors showed that the introduction of orange pomace increased the fiber content from 2.1% (for the control bread) to 4.2%. The relatively high SDF in breads with fruit pomace is probably related to the high pectin content, reaching 0.6% fresh weight in raspberries [66].

The content of protein and fat in raspberry pomace dried using various methods was at a similar level. Based on the results of the ANOVA and Tukey's test ($p > 0.05$), there were no statistically significant differences between the FD and CD samples. The amount of IDF, SDF, and TDF (soluble, insoluble, and total fiber), ash, and the energy value were significantly higher in the freeze-dried pomace (IDF by 2.44%, SDF by 10.18%, TDF by 4.50%, ash by 3.02%, and EV by 1.73%). The carbohydrate content was higher by 4.32% in the convection-dried pomace. Golovinskaia et al. [67] indicated that in raspberry fruit (bracket variety), total lipid (fat) is 0.65 g/100 g fresh weight (f. w.), protein is 1.2 g/100 g f. w., and fiber (total dietary) is 6.5 g/100 g f. w. De Souza et al. [68] obtained similar results for proteins in red raspberry (1 g/100 g f. w.) and much lower results for lipids (0.28 g/100 g f. w.) and for ash content (0.25 g/100 g fresh weight). Li et al. [69] indicated that de-oiled red raspberry pomace dried at 40 °C for 48 h contains 5.07% dry weight (d. w.) of fat, 2.58% d. w. of crude protein, and 1.3% d. w. of ash. Krivokapić et al. [70] reported that raspberry pomace contains 77.5% of total dietary fiber compared to that found in fresh fruit.

The inclusion of raspberry pomace in the gluten-free bread recipe did not significantly affect the protein content. This is probably due to the similar protein content in rice flour (7.6 g/100 g) and raspberry pomace, which was used to replace rice flour. It can be assumed that the introduction of a larger share of raspberry pomace would probably result in an increase in the protein content. In the study by Gumul et al. [64], the authors showed a significantly lower protein content in d. b. for the control gluten-free bread—2.33%. The introduction of cherry pomace at 10% and 20% did not significantly affect changes in the protein content. However, similar to our study, a slight increase in the discussed macronutrient content was observed, up to 2.55% d. b. for bread containing 20% cherry pomace.

Gluten-free bread with an unmodified recipe (control sample) contained the least fat. The use of raspberry pomace significantly increased the content of this macronutrient. As the share of raspberry pomace increased, the fat content in the tested bread increased, but these increases were statistically insignificant. In turn, the study by Cantero et al. [40] showed that the use of 8% apple pomace in gluten-free bread caused a decrease in fat content from 3.49 to 3.11 g/100 g of bread. However, the authors used gluten-free flour of unknown composition in the control dough recipe, which was then replaced with fruit pomace. Therefore, it is not possible to fully comment on the changes noted. In turn, Gumul et al. [64] found the influence of the percentage of cherry pomace addition (10% and 20%) and the flour processing temperature (80 and 120 °C) on the fat content in bread.

The conducted ANOVA and Tukey's test ($p > 0.05$) showed that there were no statistically significant differences in the average ash content in the control bread and 5CD. The ash content in the 5FD, 10FD, and 10CD breads was statistically significantly higher than in the control bread. No results regarding the ash content in gluten-free bread enriched with fruit pomace were found in the literature on this subject. He and Lu showed that the addition of apple pomace from 0% to 4% to wheat dough increased the ash content from 0.54% to 0.85% [71]. Valková et al. [72] added 1%, 2%, 5%, and 10% of apple pomace to wheat bread and showed that the ash content increased from 0.54% to 0.95%. Torbica et al. [73] described that the ash values of the control bread and the bread with apple pomace were similar.

The conducted ANOVA and Tukey's test ($p > 0.05$) showed that there were no statistically significant differences in the average content of carbohydrates in the control bread and 5CD. The content of carbohydrates in the 5FD, 10FD, and 10CD breads was statistically significantly lower than in the control bread. The available literature reports do not contain any results regarding the content of carbohydrates in gluten-free bread with fruit pomace. Valková et al. [72] showed that as the content of apple pomace increased, the value of carbohydrates increased, from 61.65% to 67.35%. Torbica et al. [73] reported that the value of carbohydrates for the control bread was 41.6 g/100 g and for bread with apple pomace was 42.2 g/100 g.

The conducted ANOVA and Tukey's test ($p > 0.05$) showed that there were no statistically significant differences in the average energy value. There are no results regarding the energy value of gluten-free bread with the addition of fruit pomace in the available literature reports.

Valková et al. [72] indicated that there were statistically significant differences between the control bread and breads with the addition of pomace, and the highest energy value was recorded for the control bread. Torbica et al. [73] reported that the energy values of the control bread and the bread with apple pomace were similar.

3.3. Antioxidant Properties of Bread and Pomace

The content of polyphenols, anthocyanins, and antioxidant activity, as determined by the ABTS and DPPH methods, of the obtained gluten-free breads and raspberry pomace are presented in Table 5.

Table 5. Antioxidant properties of breads and pomace.

Product	Probe	TPC (mg GAE/ g d. m.)	ABTS (µg TE/g d. m.)	DPPH (µg TE/g d. m.)	TAC (mg Cy3-GE/g d. m.)
Bread	0	1.37 ± 0.17 ^{a}	5.61 ± 0.61 ^{a}	1.96 ± 0.07 ^{a}	n.d.
	5FD	1.82 ± 0.15 ^{b}	9.13 ± 0.64 ^{ab}	2.64 ± 0.08 ^{b}	n.d.
	10FD	2.49 ± 0.13 ^{c}	14.6 ± 1.39 ^{b}	3.85 ± 0.12 ^{d}	n.d.
	5CD	1.67 ± 0.13 ^{ab}	9.37 ± 0.86 ^{ab}	2.68 ± 0.06 ^{b}	n.d.
	10CD	2.07 ± 0.09 ^{b}	9.14 ± 1.32 ^{ab}	3.55 ± 0.04 ^{c}	n.d.
Pomace	FD	13.7 ± 0.73 ^{A}	116 ± 1.48 ^{A}	90.2 ± 0.73 ^{A}	0.73 + 0.03 ^{A}
	CD	12.7 ± 0.67 ^{A}	111 ± 3.04 ^{B}	89.7 ± 3.18 ^{A}	0.39 + 0.02 ^{B}

n.d.—not detected; 0—control probe; 5FD—gluten-free bread with the addition of 5% freeze-dried raspberry pomace; 10FD—gluten-free bread with the addition of 10% freeze-dried raspberry pomace; 5CD—gluten-free bread with the addition of 5% convection-dried raspberry pomace; 10CD—gluten-free bread with the addition of 10% convection-dried raspberry pomace. FD—freeze-dried raspberry pomace; CD—convection-dried raspberry pomace. The values of each parameter with different superscript letters in columns are significantly different (Tukey's test, $p \leq 0.05$).

The conducted ANOVA and the post-hoc test ($p > 0.05$) showed that the average TAC and ABTS values were different for freeze-dried and convection-dried pomace. The ABTS value was 4.62% higher and the TAC value was 46.58% higher for freeze-dried pomace. Lebedev et al. [74] indicated that the TPC value was 273.9 mg GAE/100 g f. w., TAC was 37.3 mg cyan-3-G/100 g f. w., and the ABTS value was 33.2 ± 1.6 micromol TE/g f. w. for raspberry extracts, Polesie variety. De Souza et al. [68] reported that the ABTS value for red raspberry was 6.27 µmol TE/g f. w., DPPH was EC_{50} 4960.58 g f. w./g of DPPH, TPC was 357.83 mg GAE/100 g f. w., and TAC was 14.69 mg cyan-3-G/100 g f. w. Kostecka-Gugała et al. [75] freeze-dried (process parameters different from those in this study) raspberry fruits of the Polesie variety harvested in 2012 and 2013. They showed that the TPC value was 426.21, and in the following year, they found 229.60 mg chlorogenic acid per 100 g f. w., and the TAC value was 49.97. A year later, they found 45.21 mg cyan-3-G/100 g f. w, and the DPPH value, expressed as % of free radical scavenging, was 31.34, and in the following year, 41.23. Vulić et al. [76] freeze-dried raspberries (*Rubus ideaus*, cv. "Meeker") with process parameters other than those used in this work and showed that the TPC value was 2209.86 ± 70.32 mg GAE/100 g of freeze-dried raspberry, the TAC value was 144.55 ± 0.39 mg CGE/100g of freeze-dried raspberry, and the $EC_{50}^{DPPH\bullet}$ value was 0.250 mg/mL. Zorzi et al. [77] also freeze-dried raspberries with other process parameters and showed that the TAC content in the *Rubus idaeus* L. variety was 0.05 mg cyan-3-G/g fresh matter and the $EC_{50}^{DPPH\bullet}$ value was 0.6 mg/mL. The higher contents of ABTS and TAC in the freeze-dried samples were consistent with the results obtained by other researchers of the fruit drying process under various conditions [78–81]. TAC compounds are particularly sensitive to thermal drying conditions, the degradation of which occurs after the process temperature exceeds 45 °C. TPC and DPPH values did not differ statistically significantly. This is due to the greater tolerance of TPC to increased temperatures of processing processes, which only visibly reduce their share in the processed material after exceeding approximately 60 °C [81,82].

No anthocyanins were detected in the breads we tested. In the available literature reports regarding fruit pomace in gluten-free bread, the TAC value was not determined using the spectrophotometric method. Szymanowska and others [55] analyzed the anthocyanin content in waffles with the addition of freeze-dried raspberry–blueberry pomace. Changes in the tested substances accounted for up to 75%. The authors showed that the highest anthocyanin content was in waffles with the highest pomace addition. However, it should be emphasized that the baking time of the waffles was approximately 1.5 min, and the temperature was 180 °C, which did not affect the thermal degradation of anthocyanins.

The introduction of raspberry pomace increased the TPC value of gluten-free bread from 21.09% for 5CD bread to 81.75% for 10FD bread. Higher TPC values for both 5% and 10% pomace content were recorded for freeze-dried pomace. The statistical analysis performed showed that there were no statistically significant differences between the TPC values for the control bread and the 5CD bread. It was also shown that in the case of 5% addition of raspberry pomace, the average TPC value did not differ significantly between the bread to which freeze-dried pomace was added and that with the addition of convection-dried pomace. In the case of the 10% addition, the differences between the breads were statistically significant. There were also no statistically significant differences in the TPC value for 5FD and 10CD breads. After analyzing the available literature, no relevant data were found on the use of raspberry pomace in gluten-free breads and, therefore, the discussion refers to gluten-free breads with other fruit pomaces. Gumul et al. [39] showed that the TPC value in gluten-free bread with the addition of 15% apple pomace was 2050% higher than that in the control bread. Korus et al. [42] showed that the addition of 15% of blackcurrant pomace caused an increase in TPC by 139% (compared to the control group), and the addition of 15% of strawberry pomace by 1275%. The increase in the TPC value may also be caused by the Maillard reaction because its products may react with the Folin-Ciocalteu reagent [83,84]. Katina et al. [85] also indicated that the fermentation process can increase the content of total polyphenols in bread.

The use of the addition of raspberry pomace increased the antioxidant properties of gluten-free bread. The increase in the ABTS value was from 62.52% (5FD) to 159.54% (10FD), compared to bread without the addition of pomace. The statistical analysis showed that there were no significant statistical differences between the average ABTS values for bread with the addition of 5% raspberry pomace and the 10CD bread. The increase in the DPPH value was from 34.69% (5FD) to 96.43% (10FD), compared to bread without the addition of pomace. The statistical analysis showed that there were no significant statistical differences between the average DPPH values for bread with the addition of 5% raspberry pomace. Based on the analysis, it was shown that there was a correlation between the TPC value and DPPH and ABTS values. The values of the Pearson correlation coefficients were 0.96 and 0.93, respectively. Gumul et al. [39] indicated an increase in the ABTS value by 10,600% in gluten-free bread with the addition of 15% apple pomace, compared to the control bread. Cantero et al. [40] also indicated an increase in antioxidant properties in gluten-free bread with the addition of apple pomace. The DPPH value was statistically significantly higher for bread with the addition of 8% apple pomace compared to the control bread. With a lower pomace content, no differences were noted compared to the control bread [40]. Korus et al. [42] also reported that the use of pomace fruit increased the antioxidant activity. In the case of bread with 15% blackcurrant pomace, the ABTS value was higher by 39%, and for bread with the same amount of strawberry pomace, it was 371% higher compared to the control bread [42].

3.4. Principal Component Analysis (PCA)

A PCA was conducted to illustrate the similarities and differences among the samples, as depicted in Figure 2. The first principal component (PC1) accounted for approximately 69% of the overall variance, while the second principal component (PC2) explained around 18% of the variance.

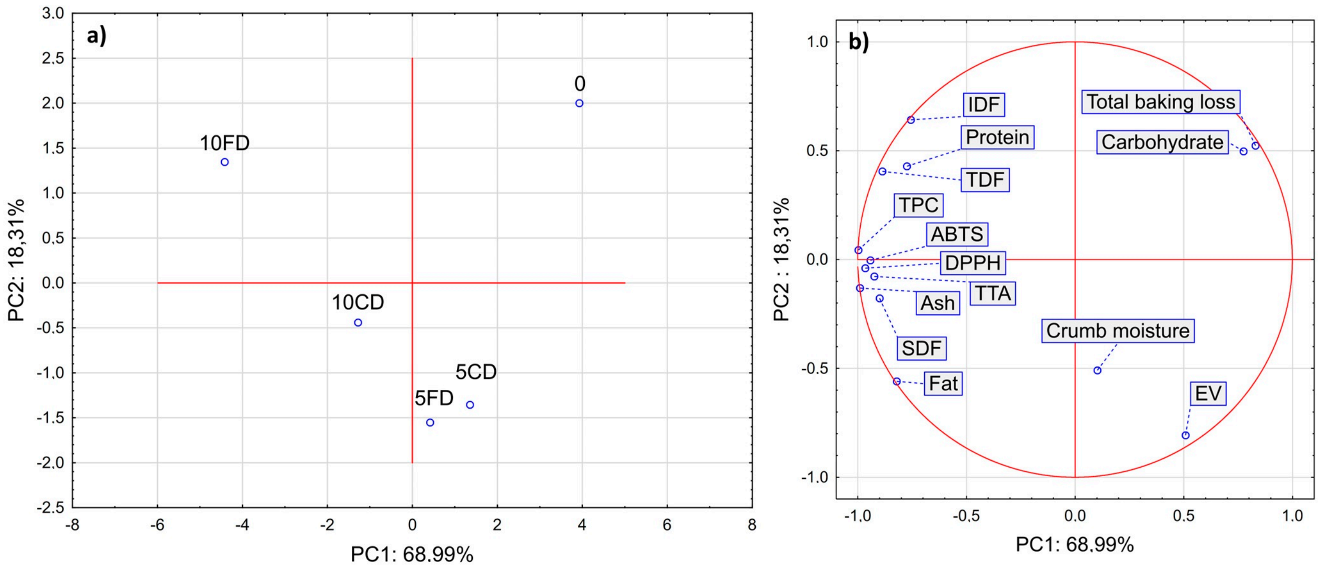

Figure 2. Principal component analysis: (**a**) projection of the samples (cases) and (**b**) projection of the variables.

The total baking loss variable was strongly negatively correlated with TPC, ABTS, DPPH, fat, ash, and SDF (Figure 2b), which means that the loss of bread mass was related to the loss of bioactive substances, fat, and soluble fiber. On the other hand, the total baking loss was strongly positively correlated with the total carbohydrate content of the bread, which means that baking losses were, to a small extent, caused by the loss of carbohydrates.

PCA showed a noticeable effect of the percentage of pomace addition on the physicochemical properties of bread. Analysis of the factor coordinates chart of the cases (Figure 2a) allowed us to distinguish three groups of points: point 0, denoting the control sample, point 10FD, denoting bread with 10% of freeze-dried pomace, and points 5CD, 5FD, and 10CD, denoting bread with 5% convection- and freeze-dried pomace content and with 10% convection-dried pomace content. The point 0 (the control sample) is located in the upper part of the coordinate system, which means a high carbohydrate content in the bread and a high baking loss coefficient. The group of three points (5CD, 5FD, and 10CD) is located in the lower part of the coordinate system, which means that these breads are characterized by a relatively high calorific value and fat content. Point 10FD (bread with 10% freeze-dried pomace content) is located in the upper left quadrant of the coordinate system, which means that it is characterized by a high content of bioactive ingredients, ash, soluble dietary fiber, and total dietary fiber. The principal component analysis confirmed our initial observations of high-quality bread with 10% freeze-dried pomace content.

4. Conclusions

The study showed that both the method of drying raspberry pomace and its percentage had a significant impact on some physicochemical properties of the tested bread. After freeze-drying, raspberry pomace was characterized by a higher content of IDF, SDF, TDF, ash, energy value, ABTS, and TAC than that dried by convection drying. Convection-dried pomace had a higher carbohydrate content.

The acidity of the bread increased statistically significantly from 2.13 mL $_{NaOH}$/10 g d. b (control bread) to 7.78 mL $_{NaOH}$/10 g d. b. (10FD). Fortifying the bread with pomace also resulted in a statistically significant reduction in the total baking loss, from 15.1% (control bread) to 10.62% (10FD). There was no statistically significant effect of the addition of raspberry pomace on the protein and water contents in the tested breads, nor on their energy values.

The addition of raspberry residues increased the content of total fiber, fat, and ash, as well as the content of antioxidant properties (TPC and DPPH). Fortifying the bread with raspberry pomace resulted in a change in the fiber content from 18.13% d. b. (control sample) up to 19.97% d. b. (10FD). Fat and ash contents in the bread increased accordingly, from 5.74% and 2.83% d. b. (control sample) to 7.18% and 3.12% d. b. (10FD). However, the sugar content decreased from 65.71% d. b. (control sample) to 63.68% d. b. (5FD).

In the case of TPC, an increase in the value of this parameter was noted from 1.37 mg GAE/g d. m. (control bread) up to 2.49 mg GAE/g d.m. (10FD), and in the case of antioxidant activity determined by the ABTS method, from 5.61 µgTE/g d. m. up to 14.56 µgTE/g d. m., and by the DPPH method from 1.96 µgTE/g d. m. up to 3.85 µgTE/g d. m., respectively.

The principal component analysis (PCA) showed that the most beneficial effect on the nutritional, bioactive, and antioxidant properties of gluten-free breads was with the 10% addition of freeze-dried raspberry pomace. In addition to nutritional quality, an important element of introducing a new product to the market is its acceptance by consumers. Thus, our future research will concern the functional properties, texture, color assessment, and sensory analysis of baked goods with the addition of raspberry pomace. The obtained results would allow the development of an appropriate recipe for gluten-free bread enriched with fruit by-products. The designed bread could be both sensory acceptable and of high nutritional quality.

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