sustainability

MDPI

Article

Bergamot Pomace Flour: From Byproduct to Bioactive Ingredient for Pasta Production

Antonio Gattuso [1,2], Alessandra De Bruno [3,*], Amalia Piscopo [1], Simone Santacaterina [1], Maria Josè Frutos [4] and Marco Poiana [1]

[1] Department of Agraria, University Mediterranea of Reggio Calabria, 89124 Reggio Calabria, Italy; antonio.gattuso@unirc.it (A.G.); amalia.piscopo@unirc.it (A.P.); simone.santacaterina@unirc.it (S.S.); mpoiana@unirc.it (M.P.)
[2] Experimental Station for the Industry of the Essential Oils and Citrus Products SSEA, 89127 Reggio Calabria, Italy
[3] Department of Human Sciences and Promotion of the Quality of Life, San Raffaele University, 00185 Rome, Italy
[4] Agro-Food Technology Department, Centro de Investigación e Innovación Agroalimentaria y Agroambiental (CIAGRO), Miguel Hernández University, 03312 Orihuela, Spain; mj.frutos@umh.es
* Correspondence: alessandra.debruno@uniroma5.it

Abstract: Contemporary consumers demonstrate an increasing preference for foods formulated with sustainable ingredients and health-promoting benefits. In this study, both demands were addressed by formulating enriched pasta using by-products derived from the processing of bergamot, a typical Calabrian citrus fruit. Wheat flour was replaced with different percentages of exhausted bergamot pomace flour (BPF: 1.5%, 2.5%, and 5%). The results indicated that bergamot pomace is a source of various phytochemical compounds, such as minerals, fibers, and polyphenols, which are beneficial to human health. The enriched pasta samples showed a significant increase in antioxidant properties, measured as a total polyphenol and flavonoid content and through chromatographic analysis. From the latter, it emerged that phenolic compounds, particularly flavonoids, were resistant to cooking. The best qualitative characteristics were shown by the sample formulated with 2.5% BPF, as also confirmed by the sensory analysis; indeed, sample C exhibited a similar level of acceptability to the control sample (A) in terms of general acceptability by the panelists. Hence, BPF can be considered as a functional ingredient for the formulation of pasta, enhancing the product's functionality, or as an addition as flour in gluten-free products.

Keywords: bergamot pomace; bioactive compounds; circular economy; citrus bergamia; citrus pomace fiber; citrus pomace flour; new formulated food; pasta; zero waste

Citation: Gattuso, A.; De Bruno, A.; Piscopo, A.; Santacaterina, S.; Frutos, M.J.; Poiana, M. Bergamot Pomace Flour: From Byproduct to Bioactive Ingredient for Pasta Production. *Sustainability* **2024**, *16*, 7784. https://doi.org/10.3390/su16177784

Academic Editor: Mariarosaria Lombardi

Received: 24 July 2024
Revised: 30 August 2024
Accepted: 5 September 2024
Published: 6 September 2024

1. Introduction

In the context of environmental sustainability and the development of strategies useful to the circular economy, it is crucial to reduce waste and generate reusable resources [1].

Agri-food waste comprises wastewater and solid residue resulting from industrial processing. The sector produces a significant number of cellulosic by-products [2], with fruit and vegetable pomace consisting largely of raw and fresh materials. For instance, in the case of citrus, pomace weight represents about 40–50% of the total processed fruits [3]. Citrus pomace (CP) constitutes a promising source of high-value compounds, including fiber, pectin, polyphenols, organic acids, cellulose, hemicellulose, macro and micro minerals, and various other products [4,5]. In recent years, the changes in environmental policy have oriented industry and research in the search for alternative solutions, combining recovery, circular economy, and resource optimization and emphasizing the added values of these by-products. This new challenge is at the heart of the 2030 Agenda [6].

Some researchers have proposed utilizing citrus residues as biomass for the production of biogas, bioethanol, and bioenergy, as a strategy to reduce the environmental

impact of their disposal [7–9]. In line with the sustainable development goals of the 2030 Agenda, the extraction of compounds from fruit processing waste is assumed as a key research line [10]. Citrus fruits are rich in fiber, polysaccharides, and phenolic components, of which insoluble dietary fiber is the main component [11]. Citrus fiber is of superior quality compared to cereal fiber, having a higher content of total dietary fiber and better features like water holding capacity and water swelling capacity due to the presence of hydroxyl groups in cellulose creating stable hydrogen bonds with water [12]. Citrus fiber can be divided in two groups: insoluble and soluble dietary fiber. The insoluble fraction is constituted by lignin, hemicellulose, and cellulose, and the soluble fraction by non-cellulosic and hemicellulose polysaccharides as well as pectin. In recent years, several innovative and green fiber extraction techniques, such as enzyme-assisted extractions [13,14], steam explosion [15], extrusion, and high hydrostatic pressures [16] have been investigated, highlighting better release, accessibility, and improved extraction efficiency. The European Food Safety Authority (EFSA) and the Food and Agriculture Organization of the United Nations (FAO) recommend a daily fiber intake of 25 g per day [17] due to its importance in human health, for instance in controlling hypertension and cardiovascular and glycemic disease [18,19] and for lowering cholesterol levels and blood pressure [20]. Moreover, fiber intake promotes intestinal microbiota activity [21], acting as prebiotics [22]. Furthermore, fiber increases the bio-accessibility of polyphenols through the binding of phenols, protecting them during digestion [23].

Polyphenols are present in the diet as individual or complex molecules, based on the degree of polymerization, and can be glycosylated or in combination with lipids, phenols, acids, or amines [24]. The effect of these compounds on human health is well known and studied. Indeed, numerous effects have been studied and reported in the literature, such as neuro and cardio protective, antidiabetic, antiaging, and anticancer effects, among others [25].

Due to these health benefits, citrus by-products are considered a potential natural source of high added value compounds with significant biological activity. Therefore, consumers' interest in eating foods rich in these compounds has recently grown. Polyphenols and fiber have been studied as ingredients in the formulation of functional foods.

In this context, research on the use of natural compounds and components with functional properties in various food types has significantly increased. This is of great interest to the food industry, enabling the production of unique products with high added value [26–28]. In addition, fiber has been considered as a functional ingredient; for example, citrus fiber has been used due to its capacity to bind water in low-fat meat products, has been suggested as fat alternative [12,29], has been used in ice cream and biscuits due to its technological and physicochemical properties [30,31], and has been an ingredient in gluten-free bread, increasing water holding capacity (WHC) and resulting in a subsequent reduction in firmness [32].

The objective of this work is to create value from bergamot pomace (BP), a by-product resulting from the processing of bergamot (*Citrus bergamia* Risso) fruit. The growing interest in this fruit has led to an increase in its production, and industrial processing of this fruit has resulted in large amounts of waste. This research adopts a perspective of total recovery and circular economy. While antioxidant compounds can be extracted from bergamot pomace [33] and used as natural antioxidants in various applications, the remaining solid residue still represents a significant amount of waste, which could be valorized for other potential uses within the food system. Consequently, this study explores the possibility to transform bergamot pomace into a flour, to use as ingredient, by leveraging the functional and technological potential of its fiber and antioxidant compounds in pasta production.

2. Materials and Methods

2.1. Raw Materials

Bergamot pomace (BP) was gathered from a local citrus processing facility located in Reggio Calabria (Calabria, Italy). BP was transferred to the Agricultural Department at

the Food technology laboratory of the University of Reggio Calabria and immediately dried to 13% moisture content in a tangential air-flow cabinet (Scirocco model, produced by Società Italiana Essicatoi, Milan, Italy) equipped with an automatic air moisture and temperature control system. Dried BP was ground into bergamot pomace flour (BPF), which was then sieved with 0.8 mm sieves (Figure 1).

Figure 1. Schematic overview of experimental plan.

2.2. Pasta Preparation

Four formulations of pasta were produced (Table 1 and Figure 1). The control sample (A) was prepared by kneading durum wheat flour (DWF—labelled as follows: protein 11 g; fats 1.0 g; carbohydrates 70 g; sugars 1.0 g; fiber 2.5 g) and water in a kneading machine (Sigma, model CHEF 20, Torbole Casaglia (Bs)—Italy). In the other three formulations, 5, 2.5, and 1.5% (DWF basis) BPF was added to the B, C, and D formulations, respectively. The dough was kept in the refrigerator for 30 min and then rolled out to a thickness of 2 mm using an electric dough sheeter (Sigma, vertical dough sheeter T50, Torbole Casaglia (Bs)—Italy) and cut into pieces measuring 7 cm in length and 3 cm in width. The pasta was cooked in a pot in boiling water with a ratio of dough/water of 1:10 (*w*/*w*). The cooking method was optimized in the laboratory; pasta samples during cooked were monitored every 30 s until the disappearance of the white core of the pasta.

Table 1. Pasta sample formulation.

Samples	DWF (g)	H_2O (g)	BPF (g)
A	500	210	-
B	475	210	25
C	487.5	210	12.5
D	492.5	210	7.5

2.3. Characterization of Bergamot Pomace Flour (BPF)

2.3.1. Determination of Color, pH, Moisture Content (MC), and Water Activity (a_w)

Color was determined using a Minolta CM-700d (Minolta, Osaka, Japan) spectrophotometer, with measurements based on the CIE L* a* b* color space (where L* indicates brightness, a* denotes redness for positive values and greenness for negative values, and b* represents yellowness for positive values and blueness for negative values). Measurements were taken directly on the flour. These coordinates were then utilized to calculate Chroma (C*) and hue angle (h°). Chroma (C*) indicates color saturation, while hue angle (h°) is defined as follows: 0°/360° for red/magenta, 90° for yellow, 180° for green, and 270° for blue or purple, thus illustrating the proportional distribution of red and yellow hues.

C* and h° were calculated as follows:

$$C^* = (a^2 + b^2)^{1/2}$$

$$h° = \arctan (b^*/a^*)$$

The pH was analyzed following the AACC International Method [34], which involved mixing 15 g of BP with 10 mL of distilled water for 30 min and then allowing it to stand for 10 additional minutes at room temperature. pH measurement was performed on the supernatant using a pH meter Crison Basic 20 (Crison Barcelona, Barcelona, Spain).

The moisture content (MC) was determined on 5 g of BP in a Sartorius Moisture Analyzer MA37 thermal balance at 105 °C. The results were expressed as % of MC.

a_w was determined at room temperature (25 °C) with a hygrometer (Aqualab LITE, Decagon, Nelson Court, Pullman, Washington, DC, USA), placing the sample into a container and then in the cell of the instrument for the analysis.

2.3.2. Determination of Nutritional Profile and Oxidative Properties of BPF

The determination of the fiber fraction was conducted by analyzing neutral detergent fiber (NDF), acid detergent fiber (ADF), and acid detergent lignin (ADL) content following the method reported by Van Soest et al. [35]. Neutral detergent soluble (NDS) was estimated as the difference between 100 and the NDF. Hemicellulose (HE) content was calculated by subtracting the NDF from the ADF, and cellulose (CE) content by taking the difference between the ADF and the residue remaining after digestion according to Trujillo et al. [36]. Crude protein (CP) was also determined following the AOAC method 984.13 [37].

Mineral content analysis was performed with inductively coupled plasma mass spectrometry (ICP-MS), using model Shimadzu MS-2030 (Shimadzu, Kyoto, Japan), as described by Botella-Martínez et al. [38]. Briefly, the calibration of single elements in the ICP-MS was conducted for mineral analysis in BPF. The operating conditions were 0.70 L min^{-1} of carrier gas, 9.0 L min^{-1} of plasma gas, and 1.10 L min^{-1} of auxiliary gas, using a radio frequency of 1.2 kW and an energy filter of 7.0 V.

The oxidative stability of BPF was studied with a OXITEST system (Accelerated Storage Test) following the method reported by Gattuso et al. [39]. A total of 45 g of BPF was submitted to an oxidation test (oxygen at 6 bar pressure; reactor temperature at 90 °C) in the OXITEST reactor to detect the Induction Period, reported as IP. IP measures the time to attain an oxidation endpoint associated with an identifiable rancidity or change in oxidation rate. The procedure was performed as described by the AOCS International Standard Procedure [40] for the determination of the oxidation stability of food, fats, and oils.

2.4. Characterization of the Physicochemical Properties of Pasta Samples

Except for the sensory analysis, all the analyses in this section were conducted on samples both before (raw) and after cooking (cooked).

Color parameters (L*, a*, b*, C*, and h°), MC, pH, and a_w were determined as reported in Section 2.3.1. Color measurements were carried out directly on the surface of the pasta at twenty casual points. For pH determination, 15 g of the product was homogenized with 100 mL of deionized water and stirred for 30 min at room temperature. Subsequently, the suspension was allowed to stand for fifteen minutes until a visible phase separation occurred.

2.5. Sensorial Analysis

The sensory analysis was conducted in accordance with ISO 13299:2003 [41] in order to assess differences among the different functionalized samples compared to the control (A sample). The test was carried out in a sensory laboratory according to ISO 8589:2007 [42] by 18 judges composed of 10 females and 8 males, with ages ranging between 25–55 years, recruited among researchers and workers of the Agricultural Department of the University of Reggio Calabria. All panelists agreed to the principles of the Declaration of Helsinki, refraining from smoking, and ingesting food and drink, excluding water, prior to the test. Pasta samples were served in white polyethylene dishes, with a secret code to identify the sample, in different orders and at different times. Pasta was cooked in salt

water and was scored considering different descriptors for appearance (color intensity, presence of stains, surface integrity, and internal homogeneity per section), aroma (citrus, cooked, off-odors, alcoholic, and oily), flavor (vegetable, pasty, bitter, salty), texture (stickiness, nerve, tooth/alto adhesiveness, granularity). In addition, other attributes such as attractiveness, harmony, and general acceptability were evaluated. The descriptors were evaluated using a nine-point intensity scale. Data were collected and elaborated by calculating the median of results.

2.6. Antioxidant Properties and Phenolic Composition (UHPLC-DAD) of Pasta Samples

The phenolic extraction method for raw and cooked samples was performed as described by Imeneo et al. [43]. In brief, 5 g of sample was mixed with methanol (20 mL), distilled water (2.5 mL), and hydrochloric acid (0.25 mL). The prepared mixtures were sonicated at 30 °C with 20 kHz ± 500 Hz of frequency in a Sonoplus ultrasonic bath (Series 2000.2, HD 2200.2—BANDELIN, Berlin, Germany). After 60 min of sonification, the extract was centrifuged for 8 min at 7000 rpm in a refrigerated centrifuge (Sigma 3-16KL, Osterode am Harz, Germany). The supernatant was recovered, filtered (Whatman n. 4 filter), and used to make up the volume in a 25 mL flask with a methanol/water mixture (1:10).

The total polyphenols content (TPC) was determined according to González-Molina et al. [44]. Briefly, 1 mL of the extract was added to 5 mL of deionized water and 1 mL of Folin-Ciocalteu reagent. Then, after 8 min of incubation, 10 mL of sodium carbonate (20%) was added, and the solution was made up to volume (25 mL) with distilled water. A blank was prepared, replacing the amount of sample with deionized water. The prepared samples were kept in dark conditions for two hours at room temperature. A double-beam Agilent 8453 diode-array UV–Visible spectrophotometer (Agilent Technologies, Santa Clara, CA, USA) was used to measure the sample's absorbance at 765 nm. Results were expressed as mg of gallic acid equivalent (GAE) per 100 g^{-1} of dry weight (mg GAE 100 g^{-1} dw).

Total Flavonoid Content (FC) was detected using the technique described by De Bruno et al. [45]. In short, in a flask (5 mL), 150 µL of $NaNO_2$ (5%, *w*/*v*) solution, 1000 µL of water, and 300 µL of $AlCl_3$ (10%, *w*/*v*) were mixed. After 6 min, 2000 µL of NaOH (1N) was added and kept for 6 min. Thus, the solution was made up to volume with deionized water. At the same time, a blank without sample was prepared, and the absorbance at 510 nm was detected. Data were expressed as milligrams of catechin equivalents per 100 g^{-1} of dry weight (mg CE 100 g^{-1} dw).

The extract was also analyzed with the liquid chromatographic technique in a UHPLC-DAD system following the method reported by Gattuso et al. [46] for identifying and quantifying the main phenolic compounds. A UPLC PLATINblue (Knauer, Berlin, Germany) equipped with a Photo Diode Array Detector PLATINblue (Knauer, Berlin, Germany) and column C18 (1.8 µm, 100 × 2 mm) at 30 °C was used to evaluate the extract (2 µL) phenolic composition. The flow rate was set at 0.4 mL min^{-1}. The eluents were water (UHPLC grade) acidified with formic acid (pH 3.10) A) and acetonitrile (B). The applied elution gradient is reported in Table 2.

Table 2. Elution gradient in chromatographic analysis.

	Time (min)	A (%)	B (%)	Flow (mL min^{-1})
1	0.00	95.00	5.00	0.400
2	3.00	95.00	5.00	0.400
3	15.00	60.00	40.00	0.400
4	15.50	0.00	100.00	0.400
5	20.00	95.00	5.00	0.400
6	22.00	95.00	5.00	0.400

External standards (1–100 mg L^{-1}) were used for the quantification of phenolic compounds. The method was validated by evaluating the limit of quantification (LOQ = SD × 3.3) and the limit of detection (LOD = SD × 10), defined as the lowest concentration in the standard solution with the percentage of the relative standard deviation (% RSD) ≤ 10%. The results were expressed as milligrams per 100 g^{-1} of dry weight (mg 100 g^{-1} dw).

2.7. Data Statistical Analysis

Data are reported as mean value ± standard deviation of data. The statistical analysis to assess the variance was one-way ANOVA, conducted by SPSS Software (Version 15.0, SPSS Inc., Chicago, IL, USA), applying the Tukey post hoc test at $p < 0.05$.

3. Results and Discussions

3.1. Bergamot Pomace Flour (BPF) Characterization

The physicochemical composition of BPF was determined, and the main results are reported in Tables 3 and 4. Regarding the color parameters, the BPF color parameters were 76.53 for L*, 4.49 for a*, 27.66 for b*, and 28.03 for C*, and the h° value was 80.84, indicating a yellow nuance. The pH value was 3.46, MC% was 13%, and aw was 0.407. These results were similar to those reported by Belluco et al. [47] for orange albedo [*Citrus sinensis* (L.) Osbeck] flour used as a functional ingredient.

The results of BPF fiber characterization are reported in Table 3 and are in accordance with values similar to other citrus fruit studied in the literature [48–50]. Fiber's fraction, expressed as % dw, showed the following percentages: 17.79 for NDF, 10.68 for ADF, 2.27 for ADL, 82.21 for NDS, 7.11 for HE, 8.41 for CE, and 7.79 for CP.

The mineral composition of BPF (Table 3) showed the highest concentrations in K (10.8 mg g^{-1} dw) and Ca (7.11 mg g^{-1} dw). It is worth noting the absence of the toxic heavy metals As, Cd, Pb, and Hg, as reported by Rahman and Singh [51]. Minerals are essential elements for humans, and their content in fruits depends on several factors, including soil composition, ripening period, and agronomic cultivation practices. However, the main relevant minerals (Ca, K, and Mg) presented values within the ranges reported for other citrus species [52–55].

Table 3. Fiber and mineral composition of Bergamot pomace flour.

Fiber Composition *		
% dw	NDF	17.79 ± 1.55
	ADF	10.68 ± 0.83
	ADL	2.27 ± 0.03
	NDS	82.21 ± 7.64
	HE	7.11 ± 0.54
	CE	8.41 ± 0.61
	CP	7.79 ± 0.89
Mineral Composition		
mg g^{-1} dw	Ca	7.11 ± 0.09
	Cu	na
	Fe	0.02 ± 0
	K	10.8 ± 0.13
	Mg	1.05 ± 0.01
	Mn	0.02 ± 0
	Na	0.65 ± 0.03
	Zn	0.14 ± 0.01
µg g^{-1} dw	As	na
	Cd	na

Cr	0.14 ± 0.06
Hg	na
Ni	na
Pb	na
Se	na

* NDF: neutral detergent fiber; ADF: acid detergent fiber; ADL: acid detergent lignin; NDS: Neutral detergent soluble; HE: hemicellulose; CE: cellulose; CP: crude protein; na: not available.

3.2. Pasta Characterization

Physicochemical parameters were evaluated on the pasta samples both before and after cooking. Table 4 shows the color coordinate values of pasta samples. L* values were higher in the raw samples but tended to decrease significantly after thermal treatment. A statistically significant change was observed in all samples, indicating a darkening effect after the cooking, especially in the samples. This could be due to Maillard reactions. Regarding the a* parameter, the pasta samples containing BP (B to C) showed no significant changes after cooking, unlike the control, which showed a decrease in this parameter after cooking. For the yellowness coordinate (b*), a decrease was observed in all pasta formulations after cooking, resulting in a lighter yellow color. The results suggest that the cooking process affected the color intensity (C*) of the pasta, leading to a reduction. The hue of pasta samples (h°) did not change after cooking. In general, the findings demonstrated that the cooking process and the addition of BPF led to slight changes in the color parameters.

Table 4. Physicochemical parameters of pasta samples.

	Samples	**A**	**B**	**C**	**D**	**Sign**
L*	Raw	78.77 ± 0.29 ^{c}	81.03 ± 0.67 ^{a}	81.06 ± 0.48 ^{a}	80.28 ± 0.69 ^{b}	**
	Cooked	76.57 ± 0.4 ^{a}	72.45 ± 0.67 ^{c}	74.64 ± 0.84 ^{b}	74.49 ± 0.4 ^{b}	**
	Sign	**	**	**	**	
a*	Raw	2.01 ± 0.07 ^{a}	1.5 ± 0.14 ^{c}	1.55 ± 0.14 ^{c}	1.89 ± 0.1 ^{b}	**
	Cooked	1.54 ± 0.07 ^{c}	1.82 ± 0.1 ^{a}	1.68 ± 0.21 ^{b}	1.52 ± 0.11 ^{c}	**
	Sign	**	ns	ns	ns	
b*	Raw	24.04 ± 0.66 ^{a}	19.55 ± 1.21 ^{c}	18.68 ± 0.67 ^{c}	21.55 ± 0.69 ^{b}	**
	Cooked	19.88 ± 0.81 ^{a}	17.67 ± 0.65 ^{b}	15.84 ± 0.5 ^{d}	16.51 ± 0.74 ^{cd}	**
	Sign	**	**	**	**	
C*	Raw	24.13 ± 0.66 ^{a}	19.6 ± 1.22 ^{c}	18.74 ± 0.67 ^{c}	21.63 ± 0.7 ^{b}	**
	Cooked	19.94 ± 0.81 ^{a}	17.77 ± 0.65 ^{b}	15.93 ± 0.5 ^{d}	16.58 ± 0.74 ^{cd}	**
	Sign	**	**	**	**	
h°	Raw	85.27 ± 0.22 ^{b}	85.64 ± 0.97 ^{a}	85.31 ± 0.85 ^{b}	85.04 ± 0.19 ^{b}	**
	Cooked	85.62 ± 0.34 ^{a}	84.15 ± 0.73 ^{c}	84.01 ± 0.78 ^{c}	84.78 ± 0.44 ^{b}	**
	Sign	ns	ns	ns	ns	
MC %	Raw	35.41 ± 0.38 ^{b}	36.18 ± 0.22 ^{ab}	36.67 ± 0.18 ^{a}	36.15 ± 0.1 ^{ab}	*
	Cooked	53.84 ± 0.4 ^{bc}	54.23 ± 0.29 ^{a}	53.21 ± 0.18 ^{c}	53.56 ± 0.23 ^{bc}	ns
	Sign	**	**	**	**	
pH	Raw	6.45 ± 0.04 ^{a}	4.66 ± 0 ^{d}	5.09 ± 0.03 ^{c}	5.73 ± 0.04 ^{b}	**
	Cooked	6.24 ± 0.11 ^{a}	4.57 ± 0.09 ^{d}	4.95 ± 0 ^{c}	5.5 ± 0.03 ^{b}	**
	Sign	ns	ns	*	*	
a_w	Raw	0.963 ± 0.011	0.956 ± 0.009	0.953 ± 0.008	0.953 ± 0.007	ns
	Cooked	0.975 ± 0.003	0.973 ± 0.003	0.975 ± 0.006	0.971 ± 0.005	ns
	Sign	ns	ns	ns	ns	

Data are presented as means ± SD (n = 3). Means within a column with different letters are significantly different by Tukey's post hoc test. Abbreviation: L*: brightness; a*: red/green value; b*: blue/yellow value; C*: chroma; h*: hue angle; ns, not significant, ** Significance at $p < 0.01$, * Significance at $p < 0.05$.

Regarding the MC values, the pasta samples naturally incorporated water after the cooking process (Table 4). It was observed that MC was significant ($p < 0.05$) among the raw samples but not significant after cooking ($p > 0.05$). The addition of BPF affected the pH value of the pasta, causing significant acidification in all samples, which was related to the amount of bergamot flour added to the formulation. Water activity is an important factor in food products because it can strongly influence their shelf life. The results of aw did not showed differences (ns) among raw and cooked samples. These results suggested that a_w was not influenced by the addition of the by-product. The addition of BPF at different concentrations, regardless of the percentage used, does not markedly affect a_w, indicating a similarity with MC% in showing no statistical difference among the cooked samples. As a result of cooking, aw increased slightly due to the higher moisture content, evenly between the different formulations but without showing statistical differences.

The sensory characteristics of the pasta samples were determined by a group of trained panelists, and the sensory attributes are presented in Figure 2. The fortified pasta samples differed from the control sample (A). Color intensity was higher in sample B, which contained a higher percentage of BPF. Regarding other appearance attributes, samples C and D scored about six, which was better than B in terms of surface integrity and internal homogeneity (these samples deviated by only one point from the control sample). The presence of stains was significantly higher in B. In terms of aromatic evaluations (Figure 2), a citrus scent was recorded with scalar values in B (5), C (3), and D (2), which covered, in part, the scent of baked, which was equal for all enriched samples. Flavor attribute data indicated the highest perception of vegetable, pasty, and bitterness notes in sample B, which contained the largest amount of BPF. Except for vegetable, for all the other characteristics, samples C and D showed similar values. No differences in saltiness were found among the samples. Among the textural attributes, scores of nerves, stickiness, and tooth/alto adhesiveness were higher in enriched samples. Granularity was slightly perceived in B and more in samples C and D but was not perceived in A.

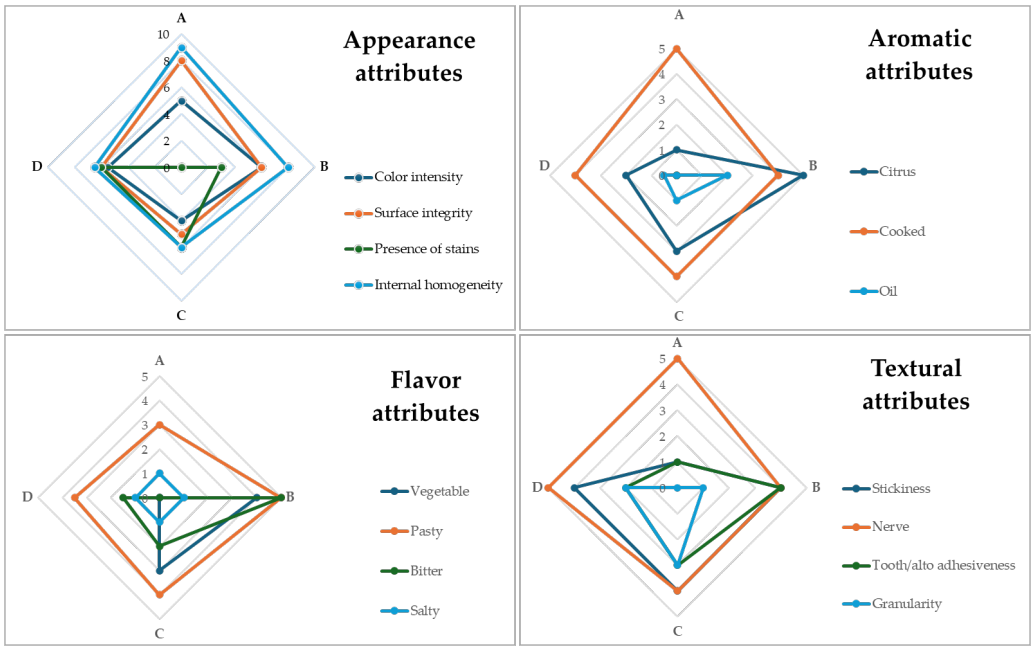

Figure 2. Sensorial attributes of pasta samples.

Moreover, the judges were asked for an evaluation based on hedonistic descriptors (attractiveness, harmony, general acceptability) (Figure 3). Even if the scores were acceptable for all samples, results clearly indicated that the sample with 1.5% BPF (D) had similar results to A.

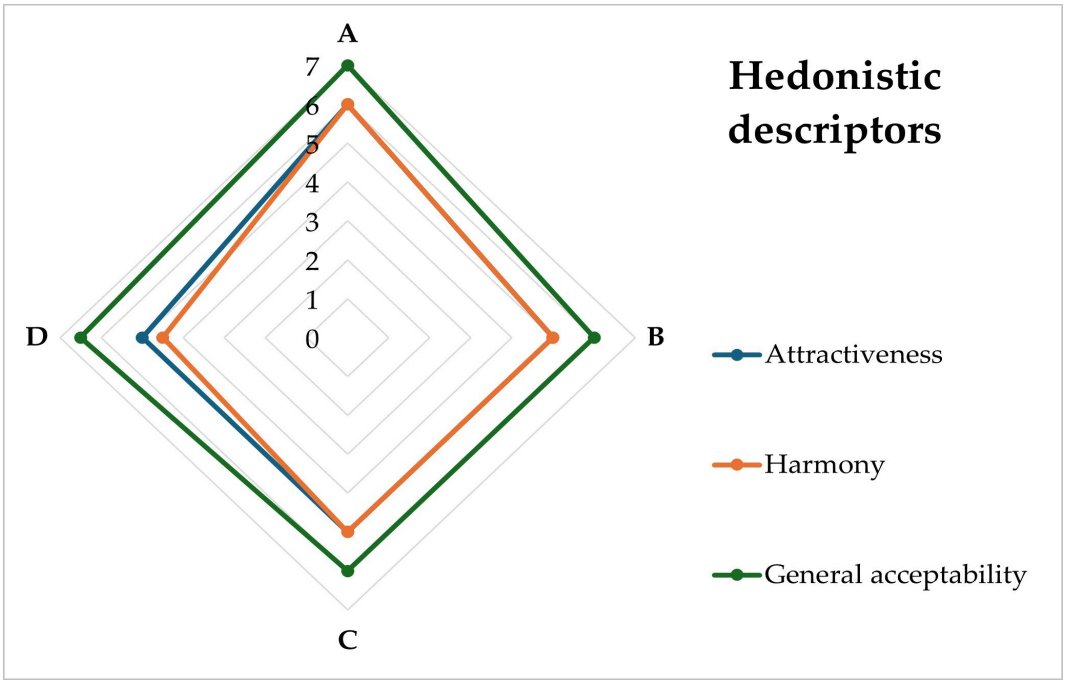

Figure 3. Hedonistic descriptors.

The Total Polyphenol Content reported in Table 5 revealed significant statistical differences ($p < 0.01$), both among the various raw and cooked pasta samples and for the same sample before and after cooking. The TPC values were statistically higher in the samples formulated with the addition of BPF compared to the control sample. The results indicated similar TPC values in raw pasta for samples formulated with a low amount of BPF (C and D). However, after cooking, all samples exhibited a substantial loss in TPC. This could be due to main factors such as the solubility of some phenolic compounds in water, especially at high temperatures, thermal degradation, oxidation reactions, or leaching into water [56,57].

Regarding TFC (Table 5) the highest content was found in enriched samples, with similar values among them. After cooking, except for sample E, no changes in TFC were detected, showing good stability.

Table 5. Antioxidant properties (TPC and TFC) of pasta samples.

	TPC (mg GAE 100 g^{-1} dw)			**TFC (mg CE 100 g^{-1} dw)**		
	Raw	Cooked	Sign	Raw	Cooked	Sign
A	36.58 ± 1.34 ^{c}	30.6 ± 0.55 ^{d}	**	12.52 ± 1.75 ^{b}	15.13 ± 2.99 ^{c}	ns
B	62.79 ± 2.92 ^{a}	50.03 ± 0.81 ^{a}	**	26.49 ± 3.52 ^{a}	28.06 ± 1.51 ^{a}	ns
C	48.62 ± 2.1 ^{b}	41.82 ± 2.24 ^{b}	**	22.73 ± 3.96 ^{a}	25.24 ± 1.66 ^{a}	ns
D	44.02 ± 1.54 ^{b}	35.08 ± 1 ^{c}	**	21.37 ± 4.03 ^{a}	21.85 ± 0.43 ^{b}	ns
Sign	**	**		**	**	

Data are presented as means ± SD ($n = 3$). Means within a column with different letters are significantly different by Tukey's post hoc test. Abbreviation: ns, not significant, ** Significance at $p < 0.01$.

After quantifying total flavonoids, we proceeded to analyze individual compounds using UHPLC. The main flavonoids detected were eriocitrin, neoeriocitrin, naringin, neohesperidin, melitidin, and brutieridin (Table 6), in accordance with those reported by Gattuso et al. [39]. The chromatographic analysis revealed the same trend reported for TFC. Specifically, after cooking, no significant changes among the flavonoids detected were observed, except in sample D for eriocitrin, melitidin, and brutieridin. All raw samples showed an

increased percentage of individual flavonoids, proportional to the added concentration of BPF. Although in most cases the increase was not significant, there was a slight rise in individual flavonoid concentrations after cooking, likely due to improved extractability.

Table 6. Individual flavonoid content of pasta samples (mg 100 g^{-1} dw).

	Eriocitrin			**Neoeriocitrin**			**Naringin**		
Samples	Raw	Cooked	Sign	Raw	Cooked	Sign	Raw	Cooked	Sign
B	0.62 ± 0.17 ^{a}	0.83 ± 0.14 ^{a}	ns	28.86 ± 3.93 ^{a}	35.66 ± 3.88 ^{a}	ns	28.09 ± 3.29 ^{a}	33.84 ± 3 ^{a}	ns
C	0.45 ± 0.08 ^{b}	0.62 ± 0.17 ^{a}	ns	14.18 ± 0.55 ^{b}	16.01 ± 0.34 ^{b}	ns	14.93 ± 0.4 ^{b}	15.92 ± 0.97 ^{b}	ns
D	0.29 ± 0 ^{c}	0.45 ± 0.02 ^{b}	**	7.04 ± 0.22 ^{c}	7.13 ± 0.28 ^{c}	ns	7.74 ± 0.02 ^{c}	8.19 ± 0.27 ^{c}	ns
Sign	**	**		**	**		**	**	
Samples	Neohesperidin			Melitidin			Brutieridin		
B	13.66 ± 1.69 ^{a}	16.48 ± 1.49 ^{a}	ns	6.33 ± 0.31 ^{a}	7.06 ± 0.45 ^{a}	ns	13.21 ± 2.84 ^{a}	16.06 ± 0.39 ^{a}	ns
C	6.15 ± 0.01 ^{b}	6.49 ± 0.22 ^{b}	ns	2.99 ± 0.13 ^{b}	3.53 ± 0.02 ^{b}	*	5.70 ± 0.23 ^{b}	6.35 ± 0.23 ^{b}	ns
D	3.14 ± 0.02 ^{c}	3.1 ± 0.03 ^{c}	ns	1.49 ± 0.04 ^{c}	1.8 ± 0.04 ^{c}	*	2.62 ± 0.18 ^{c}	3.47 ± 0.01 ^{c}	*
Sign	**	**		**	**		**	**	

Data are presented as means ± SD (n = 3). Means within a column with different letters are significantly different by Tukey's post hoc test. Abbreviation: ns, not significant, ** Significance at $p < 0.01$, * Significance at $p < 0.05$.

4. Conclusions

In this research, the effect of the addition of BPF in the quality characteristics of pasta was studied. The physicochemical characteristics (fiber, minerals) of BPF highlighted in this work, coupled with other health-promoting compounds (polyphenols, essential oil, organic acids) support its potential use in the food industry to improve the nutritional and nutraceutical properties. The addition of BPF in a food formulation such as pasta led to a natural acidification of the product, lowering the pH. This affected the sensorial and appearance attributes, particularly in the perception of the nerve and tooth/alto adhesiveness. Regarding the general acceptability preference of panelists, the results that emerged from the sensory analysis showed that the dough formulated with 2.5% BPF (C) had a similar level of acceptability to the control (A). Moreover, the antioxidant properties were improved in all the formulations considered in the experimentation. The exhausted flour also showed good values of TPC and TFC. The contribution of BPF to the dough was confirmed by chromatographic analysis (UHPLC-DAD), whereby the major flavonoids were identified and quantified.

Hence, BPF can be considered as a functional ingredient for the formulation of pasta, increasing the functionality of the product or being considered for addition as flour in gluten-free products.

Author Contributions: Conceptualization, A.G. and A.D.B.; methodology, A.G. and A.D.B., software, A.G. and A.D.B.; validation, A.P., M.J.F. and M.P., formal analysis, A.G. and S.S.; investigation, A.G. and S.S.; resources, M.P.; data curation, A.G. and A.D.B.; writing—original draft preparation, A.G. and A.D.B.; writing—review and editing, A.P., M.J.F. and M.P.; visualization, A.D.B.; supervision, A.D.B. and M.J.F.; project administration, M.P. All authors have read and agreed to the published version of the manuscript.

Funding: This work was supported by "PRIN 2017-GOOD-BY-WASTE. Obtain GOOD products exploit BY products reduce WASTE".

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: Data are contained within the article.

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

References

1. Corona, B.; Shen, L.; Reike, D.; Carreón, J.R.; Worrell, E. Towards sustainable development through the circular economy—A review and critical assessment on current circularity metrics. *Resour. Conserv. Recycl.* **2019**, *151*, 104498.
2. Torres-Leon, C.; Ramirez-Guzman, N.; Lonzono-Hernandez, L.; Martinez-Medina, G.A.; Diaz-Herrera, R.; Navarro-Macias, V.; Alvarez-Perez, O.B.; Picazo, B.; Villareal-Vazquez, M.; Ascacio-Valdes, J.; et al. Food Waste and Byproducts: An Opportunity to Minimize Malnutrition and Hunger in Developing Countries. *Front. Sustain. Food Syst.* **2018**, *2*, 52.
3. Yadav, V.; Sarker, A.; Yadav, A.; Miftah, A.O.; Bilal, M.; Iqbal, H.M.N. Integrated biorefinery approach to valorize citrus waste: A sustainable solution for resource recovery and environmental management. *Chemosphere* **2022**, *293*, 133459.
4. Nieto, G.; Fernández-López, J.; Pérez-Álvarez, J.A.; Peñalver, R.; Ros, G.; Viuda-Martos, M. Valorization of Citrus Co-Products: Recovery of Bioactive Compounds and Application in Meat and Meat Products. *Plants* **2021**, *10*, 1069.
5. Zannini, D.; Dal Poggetto, G.; Malinconico, M.; Santagata, G.; Immirzi, B. Citrus pomace biomass as a source of pectin and lignocellulose fibers: From waste to upgraded biocomposites for mulching applications. *Polymers* **2021**, *13*, 1280.
6. Smol, M.; Duda, J.; Czaplickas-Kotas, A.; Szoldrowska, D. Transformation towards circular economy (CE) in municipal waste management system: Model solutions for Poland. *Sustainability* **2020**, *12*, 4561.
7. Yu, S.; Li, L.; Zhao, H.; Tu, Y.; Liu, M.; Jiang, L.; Zhao, Y. Characterization of the Dynamic Changes of Ruminal Microbiota Colonizing Citrus Pomace Waste during Rumen Incubation for Volatile Fatty Acid Production. *Microbiol. Spectr.* **2023**, *11*, e03517–e03522.
8. Uçkun Kıran, E.; Trzcinski, A.P.; Liu, Y. Platform chemical production from food wastes using a biorefinery concept. *J. Chem. Technol. Biotechnol.* **2015**, *90*, 1364–1379.
9. Choi, I.S.; Lee, Y.G.; Khanal, S.K.; Park, B.J.; Bae, H.J. A low-energy, cost-effective approach to fruit and citrus peel waste processing for bioethanol production. *Appl. Energy* **2015**, *140*, 65–74.
10. Ademosun, A.O. Citrus peels odyssey: From the waste bin to the lab bench to the dining table. *Appl. Food Res.* **2022**, *2*, 100083.
11. Hua, M.; Lu, J.; Qu, D.; Liu, C.; Zhang, L.; Li, S.; Chen, J.; Sun, Y. Structure, Physicochemical Properties and Adsorption Function of Insoluble Dietary Fiber from Ginseng Residue: A Potential Functional Ingredient. *Food Chem.* **2019**, *286*, 522–529.
12. Jiang, Z.; Zhang, M.; Huang, Y.; Ma, C.; Mu, S.; Li, H.; Liu, X.; Ma, Y.; Liu, Y.; Hou, J. Comparison and Characterization of the Structure and Physicochemical Properties of Three Citrus Fibers: Effect of Ball Milling Treatment. *Foods* **2022**, *11*, 2665.
13. Chen, L.; Wu, Y.; Jiang, X.; Gan, D.; Fan, J.; Sun, Y.; Liu, W.; Li, X. Dietary fiber extraction from citrus peel pomace: Yield optimization and evaluation of its functionality, rheological behavior, and microstructure properties. *J. Food Sci.* **2023**, *88*, 3507–3523.
14. Fuso, A.; Viscusi, P.; Larocca, S.; Sangari, F.S.; Lolli, V.; Caligiani, A. Protease-Assisted Mild Extraction of Soluble Fiber and Protein from Fruit By-Products: A Biorefinery Perspective. *Foods* **2023**, *12*, 148.
15. Wang, C.; Lin, M.; Yang, Q.; Fu, C.; Guo, Z. The Principle of Steam Explosion Technology and Its Application in Food Processing By-Products. *Foods* **2023**, *12*, 3307.
16. Perez-Pirotto, C.; Moraga, G.; Hernando, I.; Cozzano, S.; Arcia, P. Sorption Isotherms, Glass Transition and Bioactive Compounds of Ingredients Enriched with Soluble Fiber from Orange Pomace. *Foods* **2022**, *11*, 3615.
17. Santos, D.; da Silva, J.A.L.; Pintado, M. Fruit and vegetable by-products' flours as ingredients: A review on production process, health benefits and technological functionalities. *LWT* **2022**, *154*, 112707.
18. Reynolds, A.N.; Akerman, A.; Kumar, S.; Diep Pham, H.T.; Coffey, S.; Mann, J. Dietary fiber in hypertension and cardiovascular disease management: Systematic review and meta-analyses. *BMC Med.* **2022**, *20*, 139.
19. Hojsak, I.; Benninga, M.A.; Hauser, B.; Kansu, A.; Kelly, V.B.; Stephen, A.M.; Morais Lopez, A.; Slavin, J.; Tuohy, K. Benefits of dietary fiber for children in health and disease. *Arch. Dis. Child.* **2022**, *107*, 973–979.
20. Kesbiç, O.S.; Acar, Ü.; Mohammady, E.Y.; Salem, S.M.; Ragaza, J.A.; El-Haroun, E.; Hassaan, M.S. The beneficial effects of citrus peel waste and its extract on fish performance and health status: A review. *Aquac. Res.* **2022**, *53*, 4217–4232.
21. Thomson, C.; Garcia, A.L.; Edwards, C.A. Interactions between dietary fiber and the gut microbiota. *Proc. Nutr. Soc.* **2021**, *80*, 398–408.
22. Tang, H.Y.; Fang, Z.; Ng, K. Dietary fiber-based colon-targeted delivery systems for polyphenols. *Trends Food Sci. Technol.* **2020**, *100*, 333–348.
23. Fernández-Fernández, A.M.; Dellacassa, E.; Nardin, T.; Larcher, R.; Gámbaro, A.; Medrano-Fernandez, A.; Del Castillo, M.D. In Vitro Bioaccessibility of Bioactive Compounds from Citrus Pomaces and Orange Pomace Biscuits. *Molecules* **2021**, *26*, 3480.
24. Gasmi, A.; Mujawdiya, P.K.; Noor, S.; Lysiuk, R.; Darmohray, R.; Piscopo, S.; Lenchyk, L.; Antonyak, H.; Dehtiarova, K.; Shanaida, M.; et al. Polyphenols in Metabolic Diseases. *Molecules* **2022**, *27*, 6280.
25. Rana, A.; Samtiya, M.; Dhewa, T.; Mishra, V.; Aluko, R.E. Health benefits of polyphenols: A concise review. *J. Food Biochem.* **2022**, *46*, e14264.
26. Granato, D.; Barba, F.J.; Bursać Kovačević, D.; Lorenzo, J.M.; Cruz, A.G.; Putnik, P. Functional foods: Product development, technological trends, efficacy testing, and safety. *Annu. Rev. Food Sci. Technol.* **2020**, *11*, 93–118.
27. Aziz, A.; Noreen, S.; Khalid, W.; Mubarik, F.; Niazi, M.K.; Koraqi, H.; Al-Farga, A. Extraction of bioactive compounds from different vegetable sprouts and their potential role in the formulation of functional foods against various disorders: A literature-based review. *Molecules* **2022**, *27*, 7320.
28. Sarkar, T.; Salauddin, M.; Roy, A.; Sharma, N.; Sharma, A.; Yadav, S.; Simal-Gandara, J. Minor tropical fruits as a potential source of bioactive and functional foods. *Crit. Rev. Food Sci. Nutr.* **2023**, *63*, 6491–6535.

29. Magalhães, D.; Vilas-Boas, A.A.; Teixeira, P.; Pintado, M. Functional Ingredients and Additives from Lemon by-Products and Their Applications in Food Preservation: A Review. *Foods* **2023**, *12*, 1095.
30. Mohammed, N.K.; Badrul Khair, M.F.; Ahmad, N.H.; Meor Hussin, A.S. Ice cream as functional food: A review of health-promoting ingredients in the frozen dairy products. *J. Food Process Eng.* **2022**, *45*, e14171.
31. Ali Redha, A.; Anusha Siddiqui, S.; Zare, R.; Spadaccini, D.; Guazzotti, S.; Feng, X.; Aluko, R.E. Blackcurrants: A Nutrient-Rich Source for the Development of Functional Foods for Improved Athletic Performance. *Food Rev. Int.* **2023**, *40*, 135–157.
32. Bugarín, R.; Gómez, M. Can Citrus Fiber Improve the Quality of Gluten-Free Breads? *Foods* **2023**, *12*, 1357.
33. Gattuso, A.; Piscopo, A.; Romeo, R.; De Bruno, A.; Poiana, M. Recovery of Bioactive Compounds from Calabrian Bergamot Citrus Waste: Selection of Best Green Extraction. *Agriculture* **2023**, *13*, 1095.
34. AACC. *International Approved Methods of Analysis*, 11th ed.; AACC International: St. Paul, MN, USA, 2001; 02-52.01.
35. Van Soest, P.J.; Robertson, J.B.; Lewis, B.A. Methods for dietary fiber, neutral detergent fiber, and nonstarch polysaccharides in relation to animal nutrition. *J. Dairy Sci.* **1991**, *74*, 3583–3597.
36. Trujillo, A.I.; Marichal, M.J.; Carriquiry, M. Comparison of dry matter and neutral detergent fiber degradation of fibrous feedstuffs as determined with in situ and in vitro gravimetric procedures. *Anim. Feed. Sci. Technol.* **2010**, *161*, 49–57.
37. AOAC, 1995. *Official Methods of Analysis*, 16th ed.; Association of Official Analytical Chemists: Washington DC, USA. Available online: https://www.aoac.org/ (accessed on 23 July 2024).
38. Botella-Martínez, C.; Muñoz-Tebar, N.; Lucas-González, R.; Pérez-Álvarez, J.A.; Fernández-López, J.; Viuda-Martos, M. Assessment of Chemical, Physico-Chemical and Sensory Properties of Low-Sodium Beef Burgers Formulated with Flours from Different Mushroom Types. *Foods* **2023**, *12*, 3591.
39. Gattuso, A.; Piscopo, A.; Santacaterina, S.; Imeneo, E.; De Bruno, A.; Poiana, M. Fortification of vegetable fat with natural antioxidants recovered by bergamot pomace for use as an ingredient for the production of biscuits. *Sustain. Food Technol.* **2023**, *1*, 951–961.
40. AOCS. Method Cd 12c-16. In *Official Methods and Recommended Practices of the American Oil Chemists' Society*, 6th ed.; AOCS Press: Champaign, IL, USA, 2017.
41. *ISO 13299*; Sensory Analysis—Methodology—General Guidance for Establishing a Sensory Profile. International Standardisation Organisation: Geneva, Switzerland, 2003.
42. *ISO 8589:2007*; Sensory Analysis—General Guidance for the Design of Test Rooms. International Organization for Standardization: Geneva, Switzerland, 2007.
43. Imeneo, V.; Romeo, R.; Gattuso, A.; De Bruno, A.; Piscopo, A. Functionalized Biscuits with Bioactive Ingredients Obtained by Citrus Lemon Pomace. *Foods* **2021**, *10*, 2460.
44. González-Molina, E.; Moreno, D.A.; García-Viguera, C. A new drink rich in healthy bioactives combining lemon and pomegranate. *Food Chem.* **2009**, *115*, 1364–1372.
45. De Bruno, A.; Gattuso, A.; Ritorto, D.; Piscopo, A.; Poiana, M. Effect of Edible Coating Enriched with Natural Antioxidant Extract and Bergamot Essential Oil on the Shelf Life of Strawberries. *Foods* **2023**, *12*, 488.
46. Gattuso, A.; Mafrica, R.; Cannavò, S.; Mafrica, D.; De Bruno, A.; Poiana, M. Quality Evaluation of Bergamot Juice Produced in Different Areas of Calabria Region. *Foods* **2024**, *13*, 2080.
47. Belluco, C.Z.; Mendonça, F.J.; Zago, I.C.C.; Di Santis, G.W.; Marchi, D.F.; Soares, A.L. Application of orange albedo fat replacer in chicken mortadella. *J. Food Sci. Technol.* **2022**, *59*, 3659–3668.
48. Olowu, O.; Yaman Firincioglu, S. The Nutritive value and in-vitro digestibility of peels and pomaces of different citrus species: Nutritive value assessment for citrus species by products. *J. Hell. Vet. Med. Soc.* **2023**, *74*, 6171–6179.
49. Vastolo, A.; Calabrò, S.; Cutrignelli, M.I. A review on the use of agro-industrial co-products in animals' diets. *Ital. J. Anim. Sci.* **2022**, *21*, 577–594.
50. Lashkari, S.; Taghizadeh, A. Nutrient digestibility and evaluation of protein and carbohydrate fractionation of citrus by-products. *J. Anim. Physiol. Anim. Nutr.* **2013**, *97*, 701–709.
51. Rahman, Z.; Singh, V.P. The relative impact of toxic heavy metals (THMs) (arsenic (As), cadmium (Cd), chromium (Cr)(VI), mercury (Hg), and lead (Pb)) on the total environment: An overview. *Environ. Monit. Assess.* **2019**, *191*, 419.
52. Lubinska-Szczygeł, M.; Kuczyńska-Łażewska, A.; Rutkowska, M.; Polkowska, Ż.; Katrich, E.; Gorinstein, S. Determination of the Major By-Products of Citrus hystrix Peel and Their Characteristics in the Context of Utilization in the Industry. *Molecules* **2023**, *28*, 2596.
53. Neshovska, H. Determination of the chemical and mineral composition of citrus byproducts in relation to its utilization as a feed raw material. *Bulg. J. Anim. Husb.* **2023**, *60*, 42–48.
54. Silva, J.G.S.; Orlando, E.A.; Rebellato, A.P.; Pallone, J.A.L. Optimization and validation of a simple method for mineral potential evaluation in citrus residue. *Food Anal. Methods* **2017**, *10*, 1899–1908.
55. Xu, G.H.; Chen, J.C.; Liu, D.H.; Zhang, Y.H.; Jiang, P.; Ye, X.Q. Minerals, phenolic compounds, and antioxidant capacity of citrus peel extract by hot water. *J. Food Sci.* **2008**, *73*, C11–C18.

56. Simonato, B.; Trevisan, S.; Tolve, R.; Favati, F.; Pasini, G. Pasta fortification with olive pomace: Effects on the technological characteristics and nutritional properties. *LWT* **2019**, *114*, 108368.
57. Gull, A.; Prasad, K.; Kumar, P. Nutritional, antioxidant, microstructural and pasting properties of functional pasta. *J. Saudi Soc. Agric. Sci.* **2018**, *17*, 147–153.