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Comparison of Physicochemical Properties, Volatile Profiles, and 5-Hydroxymethylfurfural and Acrylamide Content in Whole and Explosion-Puffed Wheat Grain

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Featured Application: This study highlights the potential of puffing as a processing technique to retain biologically active compounds and preserve the antioxidant activity of wheat grains, making them suitable for innovative product development. The increase in antioxidant compound content and the formation of appealing volatile aroma profiles make puffed wheat grains suitable for inclusion in breakfast cereals, snacks, and functional foods. However, the study also revealed a slight reduction in nutritional value during the husking process due to removing the grain's outer layers containing fiber, minerals, and vitamins.

Abstract: The study aimed to investigate the effects of pre-treatment (husking, sifting, and moisture adjustment) and explosion puffing on the chemical composition, volatile profile, phenolic content (free and bound), radical-scavenging activity, and formation of potentially hazardous compounds in wheat grain. Processing decreased protein, fat, ash, and dietary fiber content primarily due to removing the aleurone layer and thermal degradation leading to a diminished overall nutritional value. However, the starch content increased, along with significant changes in mono- and disaccharides, including higher maltose and glucose content attributed to starch gelatinization and hydrolysis. Thermal processing significantly altered the volatile profile, introducing new aroma-active compounds, such as pyrazines and furans, formed through Maillard and caramelization reactions. Additionally, the content of spectrophotometrically determined free phenolics and flavonoids increased, enhancing the grains' radical-scavenging potential. Safety analyses confirmed that 5-hydroxymethylfurfural (5-HMF) and acrylamide levels remained within permissible limits, ensuring compliance with food safety standards. These findings highlight the nutritional and safety implications of explosion puffing, emphasizing its potential as a wheat-processing method.

Keywords: wheat; puffing; carbohydrates; free and bound phenolics; radical-scavenging activity; volatile compounds; 5-HMF; acrylamide

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1. Introduction

Wheat is fundamental to global nutrition, serving as the primary source of carbohydrates and energy while offering significant amounts of protein, fiber, vitamins, and bioactive compounds [1]. Processing methods such as milling, fermentation, steaming, extrusion, roasting, and puffing are applied to enhance the digestibility and nutritional value of cereals [2]. Wheat grains are integral to a variety of food products, including bread, pasta, and breakfast cereals. Consumers appreciate ready-to-eat expanded grains for their convenience, nutritive value, and texture.

Puffed grains are in high demand as ready-to-eat products and ingredients for various snacks. They are commonly produced from whole grains of wheat, rice, and corn. The growing popularity of whole-grain foods is attributed to their rich content of phenolic compounds and other bioactive compounds, which offers significant health benefits [3,4]. Puffed grains are particularly nutritious because the outer layers of seeds are rich in antioxidants, minerals, and fiber [5,6]. Cereal grains, as a primary source of dietary fiber, are especially valued for their insoluble fiber content [7]. Research shows that insoluble dietary fibers slow down starch digestion more effectively than soluble fibers, contributing to health benefits [8,9].

Natural antioxidants, such as phenolics—bioactive plant-based compounds—have demonstrated positive effects on human health by reducing the risk of cardiovascular diseases, cancer, diabetes, and osteoporosis [10,11]. Moreover, volatile compounds in foods, which influence aroma and flavor, vary in type and concentration, shaping sensory attributes [12].

The production of breakfast cereals, including puffed grains, involves dehulling, moistening to facilitate starch gelatinization, and high-temperature heat treatment (puffing). At temperatures exceeding 200 °C, internal moisture is converted into superheated steam, creating pressure within the grain [13]. Due to heating, the grains soften. Once the target pressure is reached in the sealed processing vessel, it is released. The sudden drop in external pressure—while the grains retain high internal pressure—cause the trapped air within their pores to expand rapidly, making the grains "pop". This rapid expansion gives grains a light, airy texture [14], making them ready for consumption. Puffing is characterized by a controlled pressure buildup (0.1–0.3 MPa) and rapid release, preserving the color, flavor, and bioactive compounds like anthocyanins. The key factors influencing puffing include temperature, pressure, moisture content, and grain type. These processes induce changes in grain physicochemical properties, such as moisture loss, starch gelatinization, protein denaturation, and improved nutritional profiles [15]. During the Maillard reaction, which occurs at elevated temperatures between reducing sugars and amino acids or nitrogen-containing compounds, volatile compounds like pyrazines and furans are readily formed [16,17]. Additionally, bound phenolics are released during heat treatment, increasing the antioxidant capacity of grains [18,19].

High-temperature processing can lead to various adverse effects. The Maillard reaction, particularly between asparagine and reducing sugars like glucose and fructose, is known to produce acrylamide, a compound classified by the International Agency for Research on Cancer [20] as a potential human carcinogen. Similarly, the European Food Safety Authority [21,22] identifies acrylamide and furan derivatives as carcinogenic contaminants in foods [23,24]. Moreover, the conditions associated with the production of puffed grains promote both the Maillard reaction and caramelization, leading to the formation of 5-hydroxymethylfurfural (5-HMF) [25], a contaminant prevalent in numerous breakfast cereals. This compound presents potential health risks owing to its toxicity [25]. It has been reported that the high temperatures involved in the puffing process may also lead to the degradation of heat-sensitive compounds, including amino acids and fatty acids [26],

ultimately reducing the quality of proteins and fats. Heat-sensitive vitamins, such as B vitamins, are particularly susceptible [27].

Despite the wide use and nutritional potential of puffed grains, limited research exists on how processing affects the biochemical properties, antioxidant activity, volatile compounds, and the safety of wheat grains. This study aimed to compare the biochemical quality, volatile profiles, and 5-hydroxymethylfurfural and acrylamide content in whole and explosion-puffed wheat grain.

2. Materials and Methods

2.1. Materials

Certified high-purity reference standards (HPLC grade) of mono- and disaccharides, including arabinose, fructose, galactose, glycerol, glucose, inositol, xylose, lactose, maltose, mannitol, mannose, rhamnose, ribose, sucrose, sorbitol, sorbose, and trehalose, were purchased from Sigma-Aldrich Chemie Ltd. (Steinheim, Germany) and used as external standards to create calibration curves. Acetonitrile LiChoslov® (CH_3CN) was obtained from Supelco® (Bellefonte, PA, USA). Ultrapure water was produced using the PureLab Flex Elga water purification system via reverse osmosis (Veolia Water Technologies, Paris, France). Gallic acid (97.5%), Folin Ciocalteu phenol reagent, 2,2-diphenyl-1-picrylhydrazyl (DPPH) (99%), and (±)-6-hydroxy-2,5,7,8-tetrameyhylchromane-2-carboxylic acid (Trolox) (97%) were purchased from Sigma-Alrich (Buchs, Switzerland). All other chemicals used were of analytical grade.

2.2. Preparation of Puffed Grains

Whole-wheat grains (*Triticum turgidum*) were cleaned of physical impurities and then sorted by size using sieves to obtain homogeneous fractions, passing through a 10 mm sieve. The grains were husked using an AIII3-1 husking machine (Prodselmash, Novosibirsk, Russia). Following husking, the grains were sifted to remove husks, passing through a 5–6 mm sieve for further cleaning.

The cleaned grains were then moistened with warm water (35–40 °C) to adjust the moisture content to 14–14.5% and left for 14–16 h in sealed 40 L high-density polyethylene containers within the same temperature range. The required amount of water was calculated based on the initial moisture content of the raw material. To ensure uniform moisture distribution, the mixture was subjected to intensive mixing. Next, the grains were loaded into a preheated puffing gun B-35M (Jiaozuo Newest Machinery Co., Ltd., Jiaozuo, China) set to 200–220 °C. The lid was closed, and the apparatus was heated for approximately 15 min until the internal temperature reached 220–240 °C and the pressure rose to 1.2 MPa [14]. At the end of the heating period, the lid was opened, and the grains were ejected into a receiving hopper. Three independent batches were prepared for further analysis.

2.3. Determination of the Proximate Composition of Wheat and Puffed Wheat Grains

The moisture content of raw and puffed grains was determined immediately after sample grinding following the standard method AACC 44-15A [28].

The protein content was analyzed using the Kjeldhal method AOAC 991.20 [29] on a Kjeltec 2300 automatic analyzer (Foss Analytical, Höganäs, Sweden). Protein content was calculated using a conversion factor of 5.7.

The lipid content was analyzed using the Soxhlet extraction method AOAC 923.03 [30] on a SOXTEC AVANTI 2050 instrument (Foss Analytical, Höganäs, Sweden).

Ash content was determined by burning samples using gravimetric method.

The total, soluble, and insoluble dietary fiber content was analyzed by an enzymatic–gravimetric method according to the method described by Ozolina et al. [31] based on the AOAC Official Method 985.29 using Fibertec system 1010 (Foss Analytical, Höganäs, Sweden).

The carbohydrate content was calculated by subtracting fat, protein, crude fiber, and ash from the total dry solids.

Proximate composition is expressed in g per 100 g dry weight (DW).

2.4. Determination of Carbohydrates

The extraction of saccharides involved heating of the grain sample at 60 ± 1 °C for 30 min, followed by ultrasound-assisted extraction at a 50 kHz and 360 W for another 30 min at 25 ± 1 °C using an ultrasonic bath (Ultrasons, J.P. Selecta®, Barcelona, Spain). A 1.00 ± 0.01 g sample was combined with 10.0 mL of 50% aqueous acetonitrile solution (H_2O:CH_3CN v/v) in conical plastic tubes (Sarsted AG & Co. KG, Nümbrecht, Germany). After shaking with a ZX3 vortex mixer (Velp® Scientifica, Usmate Velate, Italy), the samples were centrifuged at 3169× g for 10 min at 19 ± 1 °C using a 2-16KC centrifuge (Sigma Laborzentrifugen GmbH, Osterode am Harz, Germany). Before HPLC-RID analysis, the supernatant was filtered through a hydrophilic PTFE membrane filter (Cromafil® Xtra H-PTFE, Macherey-Nagel GmbH & Co. KG, Düren, Germany). Quantitative analysis of carbohydrates was conducted using Water Alliance HPLC system (model e2695) equipped with a 2414 RI detector and a 2998 column heater (Waters Corporation, Milford, MA, USA). Carbohydrates were separated using an Altima Amino column (4.6 × 250 mm; 5 µm; Grace™, Columbia, MD, USA), with temperatures in the column and flow cell maintained at 30.0 °C, an injection volume of 15.0 µL was used, and the sample loop was washed with ultrapure water (H_2O) and acetonitrile (CH_3CN) in a 50:50 v/v ratio. The mobile phase flow rate was set at 1.0 mL/min, and data analysis was performed using Empower3 chromatography data software (build 3471) (Waters Corporation, Milford, MA, USA) [32].

Starch content was determined by the Ewers polarimetric method according to ISO 10520:2001 [33].

The content of monosaccharides, disaccharides, and starch was reported in g per 100 g DW.

2.5. Determination of Volatile Compounds

Volatile compounds were determined following the procedure described by Galoburda et al. [34]. Briefly, volatiles from the headspace above wheat and puffed wheat samples were extracted using a bipolar SMPE solid-phase microextraction fiber with an 85 µm. Carboxen/Polymethylsiloxane (CAR/PDMS) coating (Supelco Inc., Bellefonte, PA, USA) was employed. To analyze the volatile profiles in wheat and puffed wheat, 0.5 g of each sample was placed in a 20 mL vial. The extraction period included a 15-min pre-incubation period without the fiber conducted in a water bath at 40 ± 1 °C, followed by a 65-min extraction period.

The volatile compounds absorbed by the fiber were thermally desorbed in the injector of a PerkinElmer 500 gas chromatograph-mass spectrometer (GC/MS) (PerkinElmer, Inc., Shelton, CT, USA). The GC/MS was equipped with an Elite-Wax ETR capillary column (60 m × 0.25 mm i.d.; DF 0.25 µm). The GC/MS analysis parameters were set as described by Galoburda et al. [34].

Compounds were identified by matching their mass spectra against the TurboMass Nist 2008 v.2.2 mass spectral library (Perkin Elmer, Shelton, CT, USA). Additionally, linear retention indices were calculated based on the retention times of alkanes (C8-C20) and compared with values from the literature for further verification.

2.6. Determination of the Phenolics and Antiradical Activity

2.6.1. Extraction of Phenolics

The homogenized grain samples (2.0 ± 0.1 g) were extracted using an ethanol/acetone/water solution (7/7/6 $v/v/v$) in an ultrasonic bath YJ5120-1 (Cixi Tonsor Medical Instrument Co., Ltd., Zhejiang, China) at 35 kHz 10 min at 20 ± 1 °C following the method described by Tomsone & Kruma [35]. The extracts were then centrifuged at 1660× g for 5 min using a CM-6MT centrifuge (Elmi Ltd., Riga, Latvia). Residues were re-extracted using the same procedure, and the resulting supernatants were combined. The sample-to-solvent ratio was maintained at 1:10, and then triplicate extraction was performed.

Bound phenolics (BPs) were extracted following base and acid hydrolysis methods (BP–base and BP–acid extracts), as described by Alu'datt et al. [36,37]. Briefly, the residues remaining after the extraction of free phenolics were subjected to base hydrolysis using 25 mL of 0.1 M NaOH (pH 12.0) at 30 ± 1 °C for 24 h in the dark to release bound phenolics. The resulting supernatant was filtered through Whatman #1 filter paper and stored in a separate container for further analysis.

The remaining residues were then subjected to acid hydrolysis with 25 mL of 0.1 M HCl (pH 2.0) at 30 ± 1 °C for 24 h in the dark to release any remaining bound phenolics. The resulting supernatant was filtered and collected in a separate container. Each fraction obtained from base and acid extractions was analyzed in triplicate, and the values were combined to determine the total bound phenolic content.

2.6.2. Determination of the Total Phenolic Content (TPC)

The TPC of the grain extracts was determined using the Folin-Ciocalteu spectrophotometric method [38], as adapted by Tomsone and Kruma [35]. In this procedure, 0.5 mL of extract was mixed with 2.5 mL of Folin-Ciocalteu reagent (diluted 1:10 with water). After 3 min, 2 mL of sodium carbonate solution (75 g/L Na_2CO_3) was added, and the mixture was thoroughly mixed. A control sample was prepared with all reagents except the extract. The reaction mixture was incubated at 20 ± 2 °C for 30 min, after which the absorbance was measured at 756 nm. Results were calculated using a gallic acid standard curve (5–140 mg GAE/L). The TPC was expressed as mg of gallic acid equivalents (GAEs) per 100 g DW of the sample.

2.6.3. Determination of the Total Flavonoid Content (TFC)

The TFC was measured by a colorimetric method [39] with minor modifications. To 0.5 mL of extract, 2 mL of double distilled water was added, followed by 0.15 mL of 5% sodium nitrite ($NaNO_2$, 50 g/L). The extraction solution used was the same as that used for the TPC. After 5 min, 0.15 mL of 10% aluminum chloride ($AlCl_3$) solution was added, and the mixture was allowed to stand for another 5 min. Then, 1 mL of the 1 M sodium hydroxide (NaOH) was added, and the solution was mixed thoroughly. Following 15 min of incubation at room temperature, the absorbance was measured at 415 nm.

The TFC of wheat grains was expressed as mg of catechin equivalents (CE)/100 g DW.

2.6.4. Determination of DPPH Radical-Scavenging Activity

The antiradical activity of the grain extracts was assessed based on their ability to scavenge the stable 2.2-diphenyl-1-picrylhydrazil ($DPPH^{\bullet}$) radical following the method by Yu et al. [40]. The reaction was initiated by mixing 0.5 mL of grain extract with 3.5 mL of freshly prepared $DPPH^{\bullet}$ methanol solution (0.004 g DPPH in 100 mL methanol). The mixture was incubated in the dark at room temperature for 30 min, and the absorbance was measured at 517 nm [35]. The radical-scavenging capacity was expressed as millimolar

Trolox equivalents (mM TE/100 g DW of the sample). A Trolox standard curve was generated using concentrations ranging from 0.1 to 25 mM Trolox/L.

2.6.5. Determination of Ferric Reducing Antioxidant Power

The ferric reducing antioxidant power (FRAP) was evaluated using the method described by Athukorala et al. [41]. A 1 mL aliquot of the extract was mixed with 2.5 mL of phosphate buffer (200 mM, pH 6.6) and 2.5 mL of potassium ferricyanide ($K_3[Fe(CN)_6]$, 30 mM). The mixture was incubated at 50 °C for 20 min. After incubation, 2.5 mL of trichloroacetic acid (CCl_3COOH, 600 mM) was added, and the reaction mixture was centrifuged at 1660× *g* for 10 min.

The upper layer (2.5 mL) was then mixed with 2.5 mL of distilled water and 0.5 mL of ferric chloride ($FeCl_3$, 6 mM). The absorbance of the resulting solution was measured at 700 nm. The ferric reducing antioxidant power was expressed as mg ascorbic acid equivalents (AAE)/100 g DW.

2.7. Determination of the 5-HMF and Acrylamide Content

To determine the 5-HMF content, 5.0 ± 0.1 g of sample was weighed, and 50 mL of distilled water was added. The mixture was stirred using a magnetic stirrer for 20 min. The samples were centrifuged at 14,660× *g* for 10 min using a Centrifuge Pro-Research (Centurion Scientific Ltd., Stoughton, UK). The resulting supernatant was filtered using a hydrophilic PTFE membrane filter with a pore size of 0.45 µm (Macherey-Nagel GmbH & Co. KG, Düren, Germany). The filtered supernatant was then used for HPLC analysis following the methods described by Lee et al. [42] and Keke and Cinkmanis [43]. The final volume of the prepared solution was 50 mL.

Determination of 5-HMF was performed using a Shimadzu LC-40 Nexera liquid chromatograph equipped with an SIL-40C X3 autosampler and an SPD-M40 photodiode array detector (Shimadzu USA Manufacturing, Canby, OR, USA). The separation was achieved on a PerkinElmer C18 analytical column (4.6 mm × 250 mm I.D., particle size 5 µm) with the column and detector maintained at 25 °C. The mobile phase consisted of acetonitrile (HPLC grade, Sigma-Aldrich) and water (HPLC grade) in a ratio of 10:90 (*v*/*v*).

The HPLC analysis was conducted under isocratic conditions with a flow rate of 1.1 mL/min and an injection volume of 10 µL. 5-HMF detection was performed at a wavelength of 280 nm, and retention times were compared against those of a 5-HMF standard solution (HPLC grade, Sigma-Aldrich, Buchs, Switzerland). The concentration of 5-HMF in the samples was expressed in mg/kg of product.

Acrylamide content was determined according to the internal laboratory procedure PB-39/GC ed. IV of 12.01.2018 in the outsourced laboratory J.S. Hamilton (Gdynia, Poland).

2.8. Statistics

All experiments were conducted in triplicate, and the results are expressed as the mean ± standard deviation. The experimental data for wheat and puffed-wheat grains were analyzed using one-way ANOVA, followed by a post hoc *t*-test performed in Microsoft Excel 2016. Statistical significance among the parameter values was determined at a confidence level $p \leq 0.05$.

3. Results and Discussion

3.1. Effect of Processing on the Chemical Compositions of Wheat Grains

The content of proteins, fats, carbohydrates, dietary fibers, and ash in the analyzed grain samples is presented in Table 1.

Table 1. The chemical compositions of whole and puffed-wheat grains.

Variables	Content, g/100 g DW	
	Wheat Grains	Puffed Wheat Grains
Moisture	9.10 ± 0.11 [a]	5.90 ± 0.15 [b]
Protein	15.09 ± 0.24 [a]	13.70 ± 0.04 [b]
Fat	1.54 ± 0.09 [a]	0.98 ± 0.06 [b]
Carbohydrates	66.07 ± 0.11 [b]	72.20 ± 0.07 [a]
Crude fiber	15.37 ± 0.09 [a]	11.82 ± 0.11 [b]
Ash	1.93 ± 0.06 [a]	1.30 ± 0.02 [b]

Note. Average (n = 3) ± standard deviation. Different letters in the same row indicate significant differences at $p \leq 0.05$.

The findings indicate that the puffing process applied to wheat grain resulted in a 9.2% reduction in the protein content, which is correlated with the loss of protein-rich components such as the aleurone layer [44]. Furthermore, the production of puffed grain necessitates exposure to high temperatures, which generally impacts both the quality and quantity of amino acids, particularly resulting in the degradation of heat-sensitive amino acids [26]. Research indicates that the puffing process of wheat grains results in a loss of approximately 36.4% of the initial fat content. This loss is particularly linked to the decomposition of fats into free fatty acids and monoglycerides, as reported by Huang et al. [45] and Aung et al. [46]. Another credible explanation has been provided by Kaur et al. [47], revealing the formation of an amylose–fat complex that appeared after the release of free fatty acids. Along with the loss of proteins and fat, which typically diminished the quality of puffed wheat grains [48], the puffing process has been found to decrease the dietary fiber content by 23.1% and to result in a loss of minerals by 32.6% [49]. As the proportion of dietary fiber decreased, there was a corresponding increase in the content of less significant compounds, particularly carbohydrates such as starch, maltose, and glucose [47]. This increase was quantified at 9.3%. The results of the analysis of mono- and disaccharides, starch, and soluble and insoluble dietary fibers are presented in Table 2.

Table 2. The content of mono- and disaccharides, starch, and soluble and insoluble dietary fibers in whole and puffed wheat grains.

Variables	Content, g/100 g DW	
	Wheat Grains	Puffed Wheat Grains
Starch	64.40 ± 0.1 [b]	71.17 ± 0.1 [a]
Mono- and disaccharides, incl.	1.67 ± 0.09 [a]	1.03 ± 0.02 [b]
xylose	–	0.06 ± 0.01
arabinose	–	–
fructose	0.04 ± 0.01 [b]	0.35 ± 0.01 [a]
glucose	0.07 ± 0.01 [b]	0.11 ± 0.01 [a]
sucrose	1.00 ± 0.03 [a]	0.20 ± 0.01 [b]
maltose	0.14 ± 0.01 [b]	0.19 ± 0.01 [a]
lactose	–	–
unknown	0.40 ± 0.14 [a]	0.12 ± 0.01 [b]
Total dietary fiber (TDF), incl.	15.37 ± 0.09 [a]	11.82 ± 0.11 [b]
insoluble dietary fiber (IDF)	11.72 ± 0.1 [a]	8.41 ± 0.63 [b]
soluble dietary fiber (SDF)	3.65 ± 0.1 [a]	3.41 ± 0.37 [a]

Note. Average (n = 3) ± standard deviation. "–" indicates not detected. Different letters in rows indicate significant differences between samples ($p \leq 0.05$).

Removing the outer layers and subsequent thermal treatment (puffing) of wheat grains increased the relative starch content in the final product. According to Oghbaei and Prakash [2], the processing of whole wheat grains into groats increases the starch content by 22% while reducing the dietary fiber content by 61%.

As shown in Table 2, the content of insoluble dietary fiber in puffed wheat grains was significantly lower than that in whole grains. The reduction in insoluble dietary fibers, which play a crucial role in intestinal peristalsis, is a negative consequence of processing—debranning, dehulling, and thermal processing [2]. However, according to Liu et al. [50], the content of soluble dietary fibers, such as beta-glucans, which contributes to lowering cholesterol levels and improving glucose absorption, remains unchanged.

The study revealed a notable decrease in the total content of mono- and disaccharides present in puffed wheat grains. Conversely, there was an increase in the levels of maltose and glucose, which are both products of starch degradation. This trend underscores the impact of high-temperature processing on the structural alterations within grains, starch in particular. These results align with findings from other researchers [51] who studied the processes of starch gelatinization, degradation, and swelling during high-temperature grain processing.

3.2. Effect of Processing on the Volatile Compound Profiles of Wheat Grains

As a result of the study on the composition of volatile compounds in whole and puffed wheat grains, 21 and 23 volatile compounds were identified, respectively. These compounds were grouped into the following organic classes: hydrocarbons, alcohols, phenolics, ethers, aldehydes, carboxylic acids, esters, heterocyclic, nitrogen-containing compounds, and other compounds. Significant differences in the composition and quantity of volatile compounds were observed between whole and puffed wheat grains (Figure 1).

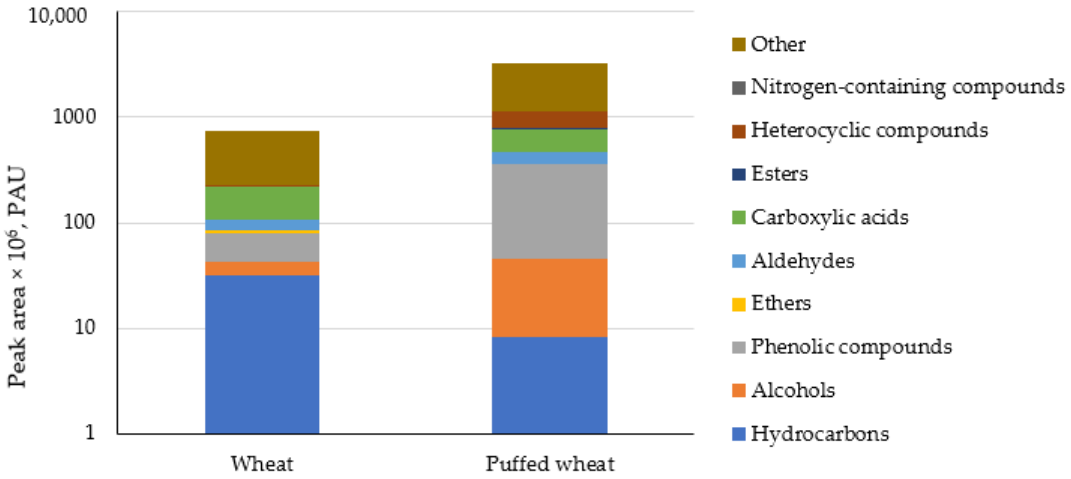

Figure 1. The content of volatile compounds in whole and puffed wheat grains.

In puffed wheat, a significant decrease in the content of hydrocarbons such as tridecane, tetradecane, and hexadecane was observed compared to whole wheat. The most notable increase was in furfural, having bread, almond, and sweet aromas. Additionally, there was an increase in the levels of heptanoic, octanoic, and nonanoic acids, which are generally described as having sweat, green, and fat aromas. In the phenolic and heterocyclic compound groups, new compounds were formed, including methylpyrazine (which imparts popcorn aroma), 2,5-dimethylpyrazine (associated with roasted, cocoa aroma), and eugenol (which has clove and honey aromas). New volatile compounds in puffed wheat grains may have formed due to the breakdown of proteins and interactions between amino

acids and reducing carbohydrates. During the Maillard reaction, the following processes and phenomena occur:

- formation of phenols and carbonyl compounds;
- formation of aldehydes [52,53];
- formation of pyrazines and furans upon heating, involving the reactions of reducing sugars, amino acids, proteins, and other nitrogen-containing compounds [17]; pyrazines such as methyl pyrazine and 2,5-dimethyl pyrazine contribute to the roasted aroma [54].

In puffed wheat grains, a noticeable increase in benzyl alcohol (which has a sweet, flowery aroma) was observed, possibly driven by thermal processing and associated hydrolysis processes. This is corroborated by studies from other researchers [3,34,54]. The study results showed that the content of hydrocarbons, characteristic of durum wheat varieties [52,53], significantly decreases in puffed grains due to thermo-oxidative processes occurring during puffing.

3.3. Effects of Processing on Phenolics and Antiradical-Scavenging Activity

The TPC in puffed wheat grains did not change compared to that in whole wheat (Table 3), while the content of the free phenolic fraction increased significantly. Thermal processing of the grains influenced the TFC level. Phenolics, which are tightly integrated into the cell wall by covalent bonds, are released during thermal processing, increasing the free phenolic fraction and enhancing the overall antioxidant capacity of the grains. The increase in free phenolics boosts the antioxidant activity of the grains, as free phenolics are more accessible for interacting with and neutralizing radicals [18,19]. These compounds benefit human health by reducing the risk of cardiovascular diseases, certain cancers, and other chronic diseases associated with oxidative stress [55].

Table 3. Antioxidant activity of compounds in whole and puffed wheat grains.

Variables	Wheat Grains	Puffed Wheat Grains
Total phenolic content, mg GAE/100 g DW, incl.	229.00 ± 3.68 ^{a}	239.77 ± 3.84 ^{a}
free	51.75 ± 3.08 ^{b}	130.70 ± 3.61 ^{a}
bound	177.26 ± 4.28 ^{a}	109.06 ± 4.06 ^{b}
Total flavonoid content, mg CE/100 g DW, incl.	142.26 ± 4.48 ^{b}	195.20 ± 3.96 ^{a}
free	48.52 ± 4.97 ^{b}	72.05 ± 4.75 ^{a}
bound	93.74 ± 3.99 ^{b}	123.15 ± 3.17 ^{a}
Radical-scavenging activity, mmol TE/100 g DW, DPPH method, incl.	7.76 ± 0.25 ^{b}	12.93 ± 0.39 ^{a}
free	2.50 ± 0.31 ^{b}	4.37 ± 0.60 ^{a}
bound	5.26 ± 0.18 ^{b}	8.56 ± 0.17 ^{a}
Ferric reducing antioxidant power, mg AAE/100 g DW, incl.	164.00 ± 3.99 ^{b}	484.39 ± 5.06 ^{a}
free	19.94 ± 3.39 ^{b}	211.13 ± 5.12 ^{a}
bound	144.06 ± 4.60 ^{b}	273.26 ± 4.99 ^{a}

Note. Average (n = 9) ± standard deviation. GAE—gallic acid equivalent; CE—catechin equivalent; AAE—ascorbic acid equivalent; DW—dry weight. Different letters in the same row indicate significant differences at $p \leq 0.05$.

The content of both free and bound forms of flavonoids was higher in puffed wheat grains than in whole grains. However, the bound form predominated in both samples.

Radical-scavenging activity, measured by the DPPH• method, increased in puffed wheat grains. The high TFC and the formation of compounds during high-temperature processing enhanced the DPPH radical-scavenging activity [56]. Many researchers have noted that the Maillard reaction, in addition to influencing the sensory properties of products, also affects antioxidant potential. Maillard reaction products, such as pyrazines and melanoidins, exhibit DPPH radical-scavenging activity [57].

The ferric reducing antioxidant power significantly increased in puffed wheat grains compared to whole grains. The antioxidant activity of food products is determined by their ability to neutralize free radicals through the presence of antioxidants [58]. Reductones, including phenolics and flavonoids, are the primary carriers of the reducing capacity of grains and can donate electrons to free radicals, thereby preventing oxidative damage [59].

3.4. Effects of Processing on 5-HMF and Acrylamide Content

The results indicate that the puffing process of wheat grain led to a significant increase in the content of 5-HMF, with nearly a 1300-fold rise observed in comparison to the initial value (Table 4). The increase in the content of 5-HMF as an intermediate in the Maillard reaction is linked to the increased temperatures that grains encounter during the processes of puffing and the direct dehydration of sugars, notably fructose and glucose, as part of caramelization [60]. The results showed a 5-HMF content of 622.98 ± 0.02 mg/kg in puffed wheat grains, which is significantly higher than the content of 66.6 mg/kg reported by Cattaneo et al. [5]. The difference could be linked to the differences in puffing process parameters. Research by Mesías et al. [61] found that wheat-based breakfast cereals tend to have higher 5-HMF content than rice-based breakfast cereals. The formation of 5-HMF depends on many factors such as temperature, water activity, the type of sugars present in the matrix, and protein content [18,53].

Table 4. Content of 5-HMF and acrylamide in whole and puffed wheat grains.

Variable	Wheat	Puffed Wheat Grains
5-HMF, mg/kg	0.47 ± 0.03	622.98 ± 0.02
Acrylamide, µg/kg	<20	240 ± 60

Note. Average (n = 3) ± standard deviation. 5-HMF—5-hydroxymethylfurfural.

Similarly, acrylamide, a known carcinogen, forms in carbohydrate- and protein-rich foods during heating at high temperatures (above 120 °C). The formation of acrylamide involves a reaction between reducing sugars (glucose, fructose) and the amino acid asparagine [55]. The study determined that the puffed wheat grains contained acrylamide at a concentration of 240 ± 60 µg/kg. The determined acrylamide concentration complies with the requirements of European Commission Regulation 2017/2158 [62], which states that the acceptable acrylamide concentration in wheat-based breakfast cereals should not exceed 300 µg/kg. As shown in Table 4, the acrylamide concentration in untreated wheat samples was below 20 µg/kg. The concentration of free asparagine in grains affects the concentration of acrylamide in breakfast cereals. The asparagine concentration in grains according to various studies [23,61] varies depending on the harvest year, fertilizer application, wheat variety, and storage conditions.

Both 5-HMF and acrylamide are byproducts of thermal processing, raising safety concerns. The acceptable daily doses of 5-HMF range from 80 to 100 mg/kg of body weight [22]. If a person weighs 70 kg, the acceptable daily dose should not exceed 5600–7000 mg of 5-HMF.

4. Conclusions

The pre-treatment and puffing processes have been shown to reduce the content of protein, fat, dietary fiber, and minerals by 9.2%, 36.4%, 23.1%, and 32.6%, respectively. These processes exert minimal effects on the levels of individual sugars, with the exception of sucrose. The heat treatment that wheat grains undergo during the puffing process leads to the development of a roasted-like aroma, which is associated with the accumulation of pyrazines and compounds typical of Amadori rearrangement reactions. These reactions include sugar dehydration, caramelization, and Maillard reactions. Furthermore, puffing positively affected the release of phenolics, increasing their reactivity, considering radical-scavenging potency. Notably, the presence of 5-hydroxymethylfurfural was detected at 623 mg/kg, while acrylamide was identified at a level of 240 μg/kg; however, both substances remained within permissible food safety limits. Future research initiatives should focus on digestibility. Moreover, assessing the long-term storability of puffed wheat grains will be essential in determining their practicality and commercial potential within the food industry.

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References

1. Wieser, H.; Koehler, P.; Scherf, K.A. Chemistry of Wheat Gluten Proteins: Qualitative Composition. *Cereal Chem.* **2023**, *100*, 23–35. [CrossRef]
2. Oghbaei, M.; Prakash, J. Effect of Primary Processing of Cereals and Legumes on Its Nutritional Quality: A Comprehensive Review. *Cogent Food Agric.* **2016**, *2*, 1136015. [CrossRef]
3. Heiniö, R.L.; Noort, M.W.J.; Katina, K.; Alam, S.A.; Sozer, N.; de Kock, H.L.; Hersleth, M.; Poutanen, K. Sensory Characteristics of Wholegrain and Bran-Rich Cereal Foods—A Review. *Trends Food Sci. Technol.* **2016**, *47*, 25–38. [CrossRef]
4. López-Perea, P.; Guzmán-Ortiz, F.A.; Román-Gutiérrez, A.D.; Castro-Rosas, J.; Gómez-Aldapa, C.A.; Rodríguez-Marín, M.L.; Falfán-Cortés, R.N.; González-Olivares, L.G.; Torruco-Uco, J.G. Bioactive Compounds and Antioxidant Activity of Wheat Bran and Barley Husk in the Extracts with Different Polarity. *Int. J. Food Prop.* **2019**, *22*, 646–658. [CrossRef]
5. Cattaneo, S.; Hidalgo, A.; Masotti, F.; Stuknyte, M.; Brandolini, A.; De Noni, I. Heat Damage and in Vitro Starch Digestibility of Puffed Wheat Kernels. *Food Chem.* **2015**, *188*, 286–293. [CrossRef]
6. Fardet, A. New Hypotheses for the Health-Protective Mechanisms of Whole-Grain Cereals: What Is beyond Fibre? *Nutr. Res. Rev.* **2010**, *23*, 65–134. [CrossRef]
7. Repo-Carrasco-Valencia, R.; Peña, J.; Kallio, H.; Salminen, S. Dietary Fiber and Other Functional Components in Two Varieties of Crude and Extruded Kiwicha (*Amaranthus caudatus*). *J. Cereal Sci.* **2009**, *49*, 219–224. [CrossRef]
8. Wang, K.; Li, M.; Han, Q.; Fu, R.; Ni, Y. Inhibition of α-Amylase Activity by Insoluble and Soluble Dietary Fibers from Kiwifruit (*Actinidia deliciosa*). *Food Biosci.* **2021**, *42*, 101057. [CrossRef]
9. Yang, Z.; Zhang, Y.; Wu, Y.; Ouyang, J. Factors Influencing the Starch Digestibility of Starchy Foods: A Review. *Food Chem.* **2023**, *406*, 135009. [CrossRef]
10. Deng, N.; Deng, Z.; Tang, C.; Liu, C.; Luo, S.; Chen, T.; Hu, X. Formation, Structure and Properties of the Starch-Polyphenol Inclusion Complex: A Review. *Trends Food Sci. Technol.* **2021**, *112*, 667–675. [CrossRef]

11. Mattila, P.; Pihlava, J.-M.; Hellström, J. Contents of Phenolic Acids, Alkyland Alkenylresorcinols, and Avenanthramides in Commercial Grain Products. *J. Agric. Food Chem.* **2005**, *53*, 8290–8295. [CrossRef] [PubMed]
12. Zhou, M.; Robards, K.; Glennie-Holmes, M.; Helliwell, S. Analysis of Volatile Compounds and Their Contribution to Flavor In. *J. Agric. Food Chem.* **1999**, *47*, 3941–3953. [CrossRef] [PubMed]
13. Subramani, D.; Kumaraguruparaswami, M.; Shivaswamy, M.S.; Tamilselvan, S.; Murugesan, M. Processing, Characteristics and Applications of Expanded Grains. *Ann. Univ. Dunarea Jos Galati Fascicle VI Food Technol.* **2023**, *47*, 195–218. [CrossRef]
14. Konkubaeva, N.; Juhnevica-Radenkova, K.; Radenkovs, V.; Galoburda, R. Effect of Coating on Physico-Chemical Characteristics of Puffed Wheat Grains. *Rural. Sustain. Res.* **2023**, *49*, 19–26. [CrossRef]
15. Hidalgo, A.; Scuppa, S.; Brandolini, A. Technological Quality and Chemical Composition of Puffed Grains from Einkorn (*Triticum monococcum* L. Subsp. *Monococcum*) and Bread Wheat (*Triticum aestivum* L. Subsp. *Aestivum*). *LWT* **2016**, *68*, 541–548. [CrossRef]
16. Hong, J.; Ting, M.; Yeo, Y.; Shu, D.; Ong, M.; Henry, J. Comparison of the Molecular Properties and Volatile Compounds of Maillard Reaction Products Derived from Animal- and Cereal-Based Protein Hydrolysates. *Food Chem.* **2022**, *383*, 132609. [CrossRef]
17. Lan, X.; Liu, P.; Xia, S.; Jia, C.; Mukunzi, D.; Zhang, X.; Xia, W.; Tian, H.; Xiao, Z. Temperature Effect on the Non-Volatile Compounds of Maillard Reaction Products Derived from Xylose—Soybean Peptide System: Further Insights into Thermal Degradation and Cross-Linking. *Food Chem.* **2010**, *120*, 967–972. [CrossRef]
18. Yu, Y.; Zhang, B.; Xia, Y.; Li, H.; Shi, X.; Wang, J.; Deng, Z. Bioaccessibility and Transformation Pathways of Phenolic Compounds in Processed Mulberry (*Morus alba* L.) Leaves after in Vitro Gastrointestinal Digestion and Faecal Fermentation. *J. Funct. Foods* **2019**, *60*, 103406. [CrossRef]
19. Hong, Q.; Chen, G.; Wang, Z.; Chen, X.; Kan, J. Effects of Different Thermal Processing Methods on Bioactive Components, Phenolic Compounds, and Antioxidant Activities of Qingke (Highland Hull-Less Barley). *Food Sci. Hum. Wellness* **2022**, *12*, 119–129. [CrossRef]
20. IARC. *International Agency for Research on Cancer*; IARC: Lyon, France, 1993.
21. Panel, E.; Chain, F. Scientific Opinion on Acrylamide in Food. *EFSA J.* **2015**, *13*, 4104. [CrossRef]
22. European Food Safety Authority (EFSA). Dietary Reference Values for Nutrients Summary Report. *EFSA Support. Publ.* **2017**, *14*, e15121E.
23. Ghazouani, T.; Atzei, A.; Talbi, W.; Fenu, M.A.; Tuberoso, C.; Fattouch, S. Occurrence of Acrylamide, Hydroxymethylfurfural and Furaldehyde as Process Contaminants in Traditional Breakfast Cereals: "Bsissa". *Food Control* **2021**, *124*, 107931. [CrossRef]
24. Verardo, V.; Moreno-Trujillo, T.R.; Fiorenza, M.; Garcia-Villanova, B.; Guerra-Hernandez, E.J. Journal of Food Composition and Analysis Influence of Infant Cereal Formulation on Phenolic Compounds and Formation of Maillard Reaction Products. *J. Food Compos. Anal.* **2021**, *104*, 104187. [CrossRef]
25. Lee, C.H.; Chen, K.T.; Lin, J.A.; Chen, Y.T.; Chen, Y.A.; Wu, J.T.; Hsieh, C.W. Recent Advances in Processing Technology to Reduce 5-Hydroxymethylfurfural in Foods. *Trends Food Sci. Technol.* **2019**, *93*, 271–280. [CrossRef]
26. Paucar-Menacho, L.M.; Duenãs, M.; Penãs, E.; Frias, J.; Martínez-Villaluenga, C. Effect of Dry Heat Puffing on Nutritional Composition, Fatty Acid, Amino Acid and Phenolic Profiles of Pseudocereals Grains. *Polish J. Food Nutr. Sci.* **2018**, *68*, 289–297. [CrossRef]
27. Garg, M.; Sharma, A.; Vats, S.; Tiwari, V.; Kumari, A.; Mishra, V.; Krishania, M. Vitamins in Cereals: A Critical Review of Content, Health Effects, Processing Losses, Bioaccessibility, Fortification, and Biofortification Strategies for Their Improvement. *Front. Nutr.* **2021**, *8*, 586815. [CrossRef]
28. AACC. 44-15A Moisture—Air—Oven Methods. In *Approved Methods of the American Association of Cereal Chemists*; AACC: St. Paul, MN, USA, 2000.
29. *991.20, A*; AOAC Official Method 991.20, Nitrogen (Crude) in Milk, to Animal Feed, Forage (Plant Tissue), Grain, and Oilseed Materials. AOAC: Arlington, VA, USA, 2019.
30. AOAC 923.03-1923 Ash of Flour. Direct Method. In *Official Methods of Analysis of AOAC International*; AOAC International: Maryland, MD, USA, 2019. Available online: http://www.aoacofficialmethod.org/index.php?main_page=product_info&products_id=1190 (accessed on 30 December 2024).
31. Ozoliņa, V.; Gramatiņa, I.; Kunkulberga, D. Methodology of Dietary Fibre Determination in Grain Products. *Res. Rural. Dev.* **2009**, *2009*, 115–119.
32. Radenkovs, V.; Juhnevica-Radenkova, K.; Kviesis, J.; Lazdina, D.; Valdovska, A.; Vallejo, F.; Lacis, G. Lignocellulose-Degrading Enzymes: A Biotechnology Platform for Ferulic Acid Production from Agro-Industrial Side Streams. *Foods* **2021**, *10*, 3056. [CrossRef]
33. *ISO 10520:1997*; Native Starch—Determination of Starch Content—Ewers Polarimetric Method. International Standard Organization: Geneva, Switzerland, 1997.
34. Galoburda, R.; Straumite, E.; Sabovics, M.; Kruma, Z. Dynamics of Volatile Compounds in Triticale Bread with Sourdough: From Flour to Bread. *Foods* **2020**, *9*, 1837. [CrossRef]

35. Tomsone, L.; Kruma, Z. Comparison of Different Solvents for Isolation of Phenolic Compounds from Horseradish (*Armoracia rusticana* L.) Leaves. In *Research for Rural Development*; Latvia University of Agriculture: Jelgava, Latvia, 2013; Volume 1, pp. 104–110.
36. Alu'datt, M.H.; Alli, I.; Ereifej, K.; Alhamad, M.; Al-Tawaha, A.R.; Rababah, T. Optimisation, Characterisation and Quantification of Phenolic Compounds in Olive Cake. *Food Chem.* **2010**, *123*, 117–122. [CrossRef]
37. Alu'datt, M.H.; Rababah, T.; Alhamad, M.N.; Gammoh, S.; Ereifej, K.; Al-Karaki, G.; Tranchant, C.C.; Al-Duais, M.; Ghozlan, K.A. Contents, Profiles and Bioactive Properties of Free and Bound Phenolics Extracted from Selected Fruits of the Oleaceae and Solanaceae Families. *Food Sci. Technol.* **2019**, *109*, 367–377. [CrossRef]
38. Singleton, V.L.; Orthofer, R.; Lamuela-Raventós, R.M. Analysis of Total Phenols and Other Oxidation Substrates and Antioxidants by Means of Folin-Ciocalteu Reagent. *Methods Enzymol.* **1999**, *29*, 152–178. [CrossRef]
39. Kim, D.; Weon, S.; Lee, C.Y. Antioxidant Capacity of Phenolic Phytochemicals from Various Cultivars of Plums. *Food Chem.* **2003**, *81*, 321–326. [CrossRef]
40. Yu, L.; Haley, S.; Perret, J.; Harris, M.; Wilson, J.; Haley, S. Antioxidant Properties of Bran Extracts from Akron Wheat Grown at Different Locations. *J. Agric. Food Chem.* **2003**, *51*, 1566–1570. [CrossRef] [PubMed]
41. Athukorala, Y.; Kim, K.; Jeon, Y. Antiproliferative and Antioxidant Properties of an Enzymatic Hydrolysate from Brown Alga, Ecklonia Cava. *Food Chem. Toxicol.* **2006**, *44*, 1065–1074. [CrossRef]
42. Lee, T.P.; Sakai, R.; Manaf, N.A.; Rodhi, A.M.; Saad, B. High Performance Liquid Chromatography Method for the Determination of Patulin and 5-Hydroxymethylfurfural in Fruit Juices Marketed in Malaysia. *Food Control* **2014**, *38*, 142–149. [CrossRef]
43. Keke, A.; Cinkmanis, I. Changes in α-Amylase Activity in Honey during the Freeze-Drying Process. *Agron. Res.* **2020**, *18*, 1717–1726. [CrossRef]
44. Lebert, L.; Buche, F.; Sorin, A.; Aussenac, T. The Wheat Aleurone Layer: Optimisation of Its Benefits and Application to Bakery Products. *Foods* **2022**, *11*, 3552. [CrossRef]
45. Huang, R.; Pan, X.; Lv, J.; Zhong, W.; Yan, F.; Duan, F.; Jia, L. Effects of Explosion Puffing on the Nutritional Composition and Digestibility of Grains. *Int. J. Food Prop.* **2018**, *21*, 2193–2204. [CrossRef]
46. Aung, T.; Kim, B.R.; Kim, S.; Shin, E.C.; Kim, M.J. Comparative Volatiles, Amino Acids, and Phenolic Compounds and Characteristics of Roasted Germinated Wheat (*Triticum aestivum* L.) during Beverage Preparation. *LWT* **2023**, *173*, 114412. [CrossRef]
47. Kaur, K.; Singh, N. Amylose-Lipid Complex Formation during Cooking of Rice Flour. *Food Chem.* **2000**, *71*, 511–517. [CrossRef]
48. Aprodu, I.; Banu, I. Antioxidant Properties of Wheat Mill Streams. *J. Cereal Sci.* **2012**, *56*, 189–195. [CrossRef]
49. Sun, X.; Sun, Z.; Guo, Y.; Zhao, J.; Zhao, J.; Ge, X.; Shen, H.; Zhang, Q.; Yan, W. Effect of Twin-Xuscrew Extrusion Combined with Cold Plasma on Multi-Scale Structure, Physicochemical Properties, and Digestibility of Potato Starches. *Innov. Food Sci. Emerg. Technol.* **2021**, *74*, 102855. [CrossRef]
50. Liu, T.; Wang, K.; Xue, W.; Wang, L.; Zhang, C.; Zhang, X.; Chen, Z. In Vitro Starch Digestibility, Edible Quality and Microstructure of Instant Rice Noodles Enriched with Rice Bran Insoluble Dietary Fiber. *LWT—Food Sci. Technol.* **2021**, *142*, 111008. [CrossRef]
51. Mariotti, M.; Alamprese, C.; Pagani, M.A.; Lucisano, M. Effect of Puffing on Ultrastructure and Physical Characteristics of Cereal Grains and Flours. *J. Cereal Sci.* **2006**, *43*, 47–56. [CrossRef]
52. Beleggia, R.; Platani, C.; Spano, G.; Monteleone, M.; Cattivelli, L. Metabolic Profiling and Analysis of Volatile Composition of Durum Wheat Semolina and Pasta. *J. Cereal Sci.* **2009**, *49*, 301–309. [CrossRef]
53. Kim, J.-S.; Lee, Y.-S. The Effect of PH on the Formation of Furfural Compounds in the Glucose and Fructose with Amino Acid Enantiomers in Maillard Reaction. *J. Food Sci. Nutr.* **2008**, *13*, 54–59. [CrossRef]
54. Xu, J.; Zhang, W.; Adhikari, K.; Shi, Y. Determination of Volatile Compounds in Heat-Treated Straight-Grade Fl Ours from Normal and Waxy Wheats. *J. Cereal Sci.* **2017**, *75*, 77–83. [CrossRef]
55. Willcox, J.K.; Ash, S.L.; Catignani, G.L. Antioxidants and Prevention of Chronic Disease. *Crit. Rev. Food Sci. Nutr.* **2004**, *44*, 275–295. [CrossRef]
56. Aung, T.; Kim, B.R.; Kim, M.J. Optimized Roasting Conditions of Germinated Wheat for a Novel Cereal Beverage and Its Sensory Properties. *Foods* **2022**, *11*, 481. [CrossRef]
57. Sun, L.; Zhuang, Y. Characterization of the Maillard Reaction of Enzyme-Hydrolyzed Wheat Protein Producing Meaty Aromas. *Food Bioprocess. Technol.* **2012**, *5*, 1287–1294. [CrossRef]
58. Gujral, H.S.; Sharma, P.; Rachna, S. Effect of Sand Roasting on Beta Glucan Extractability, Physicochemical and Antioxidant Properties of Oats. *LWT—Food Sci. Technol.* **2011**, *44*, 2223–2230. [CrossRef]
59. Omwamba, M.; Hu, Q. Antioxidant Activity in Barley (*Hordeum vulgare* L.) Grains Roasted in a Microwave Oven under Conditions Optimized Using Response Surface Methodology. *J. Food Sci.* **2010**, *75*, 66–73. [CrossRef] [PubMed]
60. Capuano, E.; Fogliano, V. Acrylamide and 5-Hydroxymethylfurfural (HMF): A Review on Metabolism, Toxicity, Occurrence in Food and Mitigation Strategies. *LWT* **2011**, *44*, 793–810. [CrossRef]

61. Mesías, M.; Sáez-Escudero, L.; Morales, F.J.; Delgado-Andrade, C. Occurrence of Furosine and Hydroxymethylfurfural in Breakfast Cereals. Evolution of the Spanish Market from 2006 to 2018. *Foods* **2019**, *8*, 158. [CrossRef]
62. Commission Regulation (EU). 2017/2158 of 20 November 2017 Establishing Mitigation Measures and Benchmark Levels for the Reduction of the Presence of Acrylamide in Food. *Off. J. Eur. Union* **2017**, *304*, 24–44.