Micronized Oat Husk: Particle Size Distribution, Phenolic Acid Profile and Antioxidant Properties

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Abstract: Oat husk (OH; hull) is a by-product generated from oat processing and is rich in insoluble fibre and phenolic compounds. The aim of this work was to study the particle size distribution, antioxidant activity, and phenolic profile of micronized OH. For this purpose, the hull was first sterilized using superheated steam and was then ground using an impact classifier mill. The particle size distribution (PSD) of the ground husk was determined using the laser diffraction method and the parameters characterizing the PSD of the ground husk, and its antioxidant activity were calculated. In addition, UPLC-MS/MS analysis of phenolic acids was also performed. Micronization of the sterilized husk effectively decreased the size of the particles, and with the increasing speed of the rotor and classifier, the median size of the particles (d50) decreased from 63.8 to 16.7 µm. The following phenolic acids were identified in OH: ferulic, caffeic, p-hydroxybenzoic, vanillic, syringic, and synapic acid. Ferulic acid constituted about 95% of total phenolic acids. The antioxidant activity of the obtained extracts increased as the particle size of the micronized husk decreased. The highest half maximal inhibitory concentration (EC50 index) was found for chelating power, and the lowest was found in the case of radical scavenging activity against DPPH.

Keywords: oat hull; micronization; impact classifier mill; microbiological purity; particle size distribution; total phenolic content; antioxidant properties

1. Introduction

Oat is becoming more popular as a raw material in various sectors of the food industry because of the well-known health benefits of its products. Oat grain is rich in many bioactive compounds such as phenolic acids, flavonoids, avenanthramides, and fibre [1,2]. Oat husk (OH; hull) is a by-product obtained from oat processing for food purposes. It is assumed that 2.75–3.3 million tons of OH is generated as a by-product [3]. OH usually represents an average of 27% of oat grain [4]. Emmons and Peterson [5] found a similar level of total phenolics in OH as in oat groat and husked oat. However, studies conducted by Piątkowska et al. [6] on different parts (e.g., husk, bran, endosperm and whole grain) of 13 oat varieties showed that OH contained the highest level of polyphenols and exhibited the highest antioxidant activity. Additionally, OH is a rich source of insoluble dietary fibre with health-promoting properties. This type of fibre includes cellulose and lignins, which are the components of plant cell walls, and absorbs water but does not dissolve in it. Dietary fibre increases the volume and softness of the stool and supports normal intestinal peristalsis [7]. The main constituent of OH is lignocellulosic fibres, which are made up of cellulose, hemicelluloses, and lignin [8]. The composition of OH is as follows: ~33% crude...
fibre, ~33% pentosans, ~13% lignin, ~4% protein, and ~5% ash [9]. OH is mainly used in animal feed or in the production of industrial solvents. However, it can also be used as an ingredient in high-dietary fibre foods [10]. Studies analysing the possibility of using OH as a food additive are limited in the literature. Piwińska et al. [11] added a mixture of OH with a soluble fraction of oat and produced wheat pasta. Oliveira et al. [8] showed that OH is a valuable source of cellulose fibres that can be used in the production of hydrogels that can be used in different applications.

Grinding is an important process that is followed in the food industry. The particle size of the ground seeds determines the properties of the final products [12,13]. Ultra-fine grinding (micronization), in particular, has become very popular in recent years [14–16]. Micronization is a process by which the particle size of a material is reduced to a nanosize range [17,18], thereby improving dispersion, chemical and biological activities, and the rate of nutritional absorption from food [19]. In fibre-rich plant materials, micronization enhances the water absorption of the particles and the solubility of fibre [20]. It also improves mouthfeel and increases the release of flavours [18]. Importantly, micronization improves the extraction of antioxidant compounds and helps in the release of many bioactive compounds bound to the food matrix [19,21,22].

Due to its high fibre content, the proper grinding of OH using traditional size reduction methods is difficult. Moreover, after conventional grinding, the OH particles produce an unpleasant, prickly mouth feeling [23]. Therefore, the fine grinding of such a material is recommended. Currently, no studies on the process of OH micronization and its properties can be found in the literature. Therefore, this work aimed to study the influence of the working parameters of an impact classifier mill on the particle size distribution (PSD), phenolic profile, and antioxidant properties of pulverized OH.

2. Materials and Methods

OH was received from AG Feeding Sp. z o.o. (Gdynia, Poland). Folin–Ciocalteu reagent, DPPH (2,2-diphenyl-1-picrylhydrazyl), gallic acid monohydrate, methanol, and ABTS (2,2-diphenyl-1-picrylhydrazyl) were purchased from the Sigma–Aldrich company (Poznań, Poland). Methanol and acetonitrile HPLC grade were purchased from Merck (Darmstadt, Germany). Formic acid LC-MS grade was purchased from Merck (Darmstadt, Germany). Ultrapure water was obtained in-house with a purification system (Milli-Q-Simplicity-185, Millipore Corp., Burlington, MA, USA). Reference standard kaempferol was purchased from Fluka AG (Buchs, Switzerland). All chemicals were of analytical grade.

2.1. Basic Composition

The moisture content (Method 925.10), crude protein content (Method 992.33; Nx6.25), ash content (Method 942.05), and total fibre content (Method 985.29) of OH were determined using standard methods [AOAC, 2011] [24].

2.2. Husk Sterilization

Before being ground, OH was sterilized using superheated steam generated from a thermal sterilizer of the authors’ own design. The sterilizer consisted of an insulated heated chamber equipped with a paddle mixer and a superheated steam supply system. The screw conveyor delivered the material to be ground to the heated chamber, the walls of which were heated up to a temperature of 200 °C. The paddles in the sterilizer chamber moved the material while mixing it. This prevented the material that came in direct contact with the chamber wall for a long time from burning. The raw supplied material was heated by the walls of the sterilizer. After preheating the material, a superheated steam that was 200 °C was supplied to the chamber, which had a total length of 6 m. The exhaust hood used at the end of the chamber removed the excess steam and did not allow the steam to retract. The total sterilization time was 4 min. The material from the sterilizer went directly into the cooling conveyor, which also had a length of 6 m. The efficiency of the sterilization
and cooling process was identical; therefore, the material that was cooled to a temperature of about 30 °C was sent directly to the mill.

2.3. Microbiological Purity of OH

The OH samples were analysed before and after sterilization for the total number of yeasts and moulds [25], the total number of microorganisms [26], coliform counts [27], and Salmonella [28] and Bacillus cereus counts [29].

2.4. Micronization of OH

The sterilized OH was micronized using an impact classifier mill equipped with a rotor disc with 12 hammers and a classifier [30]. The mill was integrated with a centrifugal fan (airflow 2 m³·s⁻¹). The OH was ground by setting the disc at four different speeds: 2600, 2970, 3340, and 3710 rpm. It was pulverized by the hammers and passed through a liner with a corrugated surface. The pulverized material was then transported through a baffle assembly into a classifier wheel, which restricts the coarse particles from escaping to outside the mill, and these particles were recirculated back into the milling chamber. For each speed of the rotor disc, six different classifier wheel speeds were applied: 480, 965, 1450, 1930, 2410, and 2890 rpm. A detailed description of the mill used in this study was given by Dziki et al. [30].

2.5. Particle Size Distribution

The PSD of the micronized OH was measured by laser light scattering from 0.01 to 3.0 mm using a laser particle size analyser (Malvern Mastersizer 3000, Malvern Instruments Ltd., Worcestershire, UK). A total of 5 g of micronized OH sample was added to the inlet chamber, and the PSD was measured automatically by laser diffraction using the dry dispersion method [31]. PSD was expressed as $d_{10}$, $d_{50}$ (median diameter), and $d_{90}$, which represent the 10th, 50th, and 90th percentile of the total volume, assuming the particles have a spherical shape. Moreover, the volume-based size distribution (Span) was calculated [32] using the following equation:

$$\text{Span} = \frac{d_{90} - d_{10}}{d_{50}}$$

2.6. Antioxidant Properties

To study the antioxidant activity, 50% methanolic (methanol: water, 1:1 v/v) extracts of micronized OH were prepared. An amount of 250 mg of plant material was triple extracted using 5 mL of 50% aqueous methanol in a laboratory shaker for 30 min and then centrifuged. The combined extracts were used for further analyses [33].

The antioxidant activity of the OH extracts was determined by three assays: chelating power (CHEL), DPPH, and the ABTS radical scavenging activity. CHEL was determined according to the method presented by Guo et al. [34] with slight modification [35]. Radical scavenging activity of the OH extract against stable DPPH and the ABTS radical scavenging activity were determined according to Brand-Williams et al. [36] and Re et al. [37], respectively. The blank samples contained 50% methanol instead of the extract.

The chelating potential was assessed according to the formula:

$$\% \text{ inhibition} = [1 - (AA/AC)] \times 100$$

Free radical scavenging activity was assessed according to the following equation:

$$\text{scavenging} \% = [\frac{(AC - AA)}{AC}] \times 100$$

where AC is the absorbance of the control, and AA is the absorbance of the sample.

The half maximal inhibitory concentration (EC₅₀) values were calculated at fitted models, as the concentration of the tested compound produced 50% of the maximum inhibition based on a dose-dependent mode of action [38].
2.7. UPLC-MS/MS Phenolics Acids Analysis

Three OH samples (200 mg each) of OH with the highest degree of fineness were selected and hydrolyzed with 4N NaOH and 2% ascorbic acid solution for 4 h at room temperature. Then, the samples were cooled in ice and were acidified using ice-cold 6M HCl to achieve a pH value of about 2. The resulting mixtures were centrifuged at 8000 rpm (Sigma 2-16) for 20 min. Supernatants of ethyl acetate were extracted three times. The organic phase was collected, filtered, and evaporated to dryness at 35 °C in a rotary evaporator. The residue was dissolved in 30% methanol and was stored in a freezer.

The phenolic acids were determined by an ACQUITY UPLC system equipped with a PDA and a triple quadrupole mass detector (TQD, Waters, Milford, MA, USA). Samples were separated on a Waters Acquity UPLC HSS C18 column (100 × 2.1 mm, 1.8 µm) with a mobile phase of solvent A (acidified water, 0.1% formic acid) and solvent B (acidified acetonitrile, 0.1% formic acid). The solvent gradient was programmed as follows: 8% to 20% B in 8 min; 20% to 95% B in 2.9 min; and 95% to 8% B in 2 min at a flow rate of 0.5 mL/min. The column temperature was maintained at 30 °C. The injection volume was set to 2.5 µL. Compounds were interpreted based on data from mass spectra. ESI ionization was performed in negative ion mode. Data processing was performed using MassLynx V4.1 software, Waters (Milford, MA, USA).

2.8. Statistical Analysis

All measurements were performed in triplicate unless otherwise noted. Analysis of variance was performed (one-way or two-way), and significant differences between means were determined using Tukey’s test. In addition, correlation and multilinear regression analyses were performed. The significance level of α was established at 0.05. Statistica 13.3 software (StatSoft, Tulsa, OK, USA) was used for the calculations.

3. Results and Discussion

3.1. Basic Composition and Microorganisms Identification

The basic chemical composition of OH was as follows: ash content 3.42 ± 0.11%, protein content 1.27 ± 0.08%, total fibre content 90.7 ± 1.23%, and initial moisture content 10.2 ± 0.15%. The microorganisms identified in the OH before and after sterilization are summarized in Table 1. Microorganisms are an integral part of cereal grains. The external parts of grains such as OH are especially susceptible to contamination during the period of grain ripening, harvesting, and storage [39]. Although microorganisms do not grow under lower water activity, bacteria and fungi can thrive for a long time, and under favourable conditions, they become active and present a potential hazard [40]. Escherichia coli, B. cereus, and Salmonella were not detected both before and after sterilization in any of the OH samples. However, moulds and yeasts were detected. The average number of moulds and yeasts and the total number of bacteria before sterilization were 4.0·10^5 and 4.6·10^6 cfu·g⁻¹, respectively. Sterilization significantly decreased the number of microorganisms to an acceptable level [41]. The numbers of yeasts and moulds were found to have been decreased below 10^1 cfu·g⁻¹, whereas the average total aerobic plate count was 2.6·10^3 cfu·g⁻¹. According to the literature data, the content of yeasts and moulds in healthy cereal grains and oilseeds does not exceed 10^4 cfu·g⁻¹ [42]. In addition, in the study, it was observed that sterilization decreased the moisture of the hull by up to 3.4 ± 0.22%. A decrease in the moisture content of the plant materials is known to increase their friability and usually results in the grinding process being more effective [43,44].

| Oat Husk            | Yeast and Moulds             | Aerobic Plate Count | Escherichia coli   | Batocillus Cereus | Salmonella       |
|---------------------|-----------------------------|----------------------|-------------------|-------------------|-----------------|
| Before Sterilization| 4.0·10^5 ± 2.0·10^5         | 4.6·10^6 ± 2.3·10^6  | nd                | nd                | nd              |
| After Sterilization | <10^1                       | 2.6·10^3 ± 0.5·10^3  | nd                | nd                | nd              |

* not detected.

Table 1. Microorganisms (cfu·g⁻¹) identified in the OH before and after sterilization.
3.2. Particle Size Distribution

Example curves of the PSD of OH are presented in Figure 1, and the parameters that describe the PSD of ground OH are summarized in Table 2. The values of \(d_{10}\) changed in a relatively narrow range from 3.2 to 7.8 µm, whereas those of \(d_{50}\) and \(d_{90}\) ranged from 16.7 to 63.8 µm and from 75.1 to 282 µm, respectively. In the case of Span, which refers to the PSD width, the values increased from 4.1 to 7.9. The degree of fineness of the fibre-rich oat flour has a significant influence on the properties of many final products, such as bread [45], pasta [46], and sausage [47]. Particle size affects the texture and mouthfeel [48] of products and increases their bioavailability [49].

![Figure 1. Example curves of the PSD of OH (sample H18, eight repetitions).](image)

Table 2. Parameters that describe the particle size distribution of micronized OH.

| Sample | Revolution of Disc (rpm) | Revolution of Classifier Wheel (rpm) | \(d_{10}\) (µm) | \(d_{50}\) (µm) | \(d_{90}\) (µm) | Span |
|--------|--------------------------|---------------------------------|----------------|----------------|----------------|-------|
| H1     | 2600                     | 480                             | 6.2 ± 0.05 m,n,s | 41.3 ± 1.02 h | 249 ± 11.4 i | 5.9 ± 0.16 i |
| H2     | 2600                     | 965                             | 6.1 ± 0.07 m   | 55.7 ± 3.76 i | 282 ± 16.0 n | 5.0 ± 0.23 s |
| H3     | 2600                     | 1450                            | 5.5 ± 0.05 l   | 50.6 ± 1.33 j | 277 ± 5.1 m,n | 5.4 ± 0.12 h |
| H4     | 2600                     | 1930                            | 5.3 ± 0.04 k   | 47.6 ± 1.45 k | 269 ± 6.91 m,n | 5.5 ± 0.04 h |
| H5     | 2600                     | 2410                            | 4.9 ± 0.04 l   | 40.0 ± 0.71 h | 264 ± 4.61 m | 6.5 ± 0.11 j |
| H6     | 2600                     | 2890                            | 4.2 ± 0.02 e   | 22.9 ± 0.24 d,e | 185 ± 3.1 f | 7.9 ± 0.11 l |
| H7     | 2970                     | 480                             | 7.1 ± 0.09 n   | 56.1 ± 3.18 h | 268 ± 10.9 l,m | 4.6 ± 0.10 c,f |
| H8     | 2970                     | 965                             | 5.1 ± 0.07 k   | 42.6 ± 1.50 h | 233 ± 3.5 h,j | 5.4 ± 0.11 h |
| H9     | 2970                     | 1450                            | 4.7 ± 0.07 h   | 40.1 ± 3.97 h | 224 ± 17.1 h | 5.5 ± 0.13 h |
| H10    | 2970                     | 1930                            | 3.8 ± 0.04 d   | 26.3 ± 0.78 f | 179 ± 7.3 f | 6.7 ± 0.10 j |
| H11    | 2970                     | 2410                            | 3.8 ± 0.07 d   | 20.8 ± 0.95 c,d | 150 ± 7.2 d | 7.1 ± 0.23 k |
| H12    | 2970                     | 2890                            | 3.4 ± 0.03 c   | 15.5 ± 0.22 a | 105 ± 4.3 b | 6.6 ± 0.21 j |
| H13    | 3340                     | 480                             | 7.7 ± 0.04 e   | 63.8 ± 1.67 k | 261 ± 4.7 k | 4.0 ± 0.06 a,b |
| H14    | 3340                     | 965                             | 4.1 ± 0.23 e   | 25.8 ± 3.98 e,f,l | 174 ± 19.9 c,d,l | 6.6 ± 0.28 j |
| H15    | 3340                     | 1450                            | 5.0 ± 0.07 f   | 41.2 ± 0.89 h | 203 ± 4.7 g | 4.8 ± 0.07 f,g |
| H16    | 3340                     | 1930                            | 4.3 ± 0.08 f   | 28.8 ± 0.81 f | 180 ± 8.0 f | 6.1 ± 0.15 i |
| H17    | 3340                     | 2410                            | 3.4 ± 0.05 b,c | 20.2 ± 0.41 b,c,d | 125 ± 2.6 c | 6.0 ± 0.11 i |
| H18    | 3340                     | 2890                            | 3.2 ± 0.03 a   | 17.4 ± 0.16 a,b | 99 ± 2.3 b | 5.5 ± 0.11 h |
| H19    | 3710                     | 480                             | 7.8 ± 0.12 a   | 67.4 ± 1.26 l | 265 ± 5.1 l,m | 3.8 ± 0.11 a |
| H20    | 3710                     | 965                             | 7.1 ± 0.09 n   | 57.1 ± 1.04 j | 243 ± 9.0 l,j | 4.1 ± 0.11 b,c,d |
| H21    | 3710                     | 1450                            | 4.5 ± 0.05 f   | 34.5 ± 0.66 g | 161 ± 3.9 d | 4.5 ± 0.09 e |
| H22    | 3710                     | 1930                            | 4.2 ± 0.04 e   | 26.2 ± 0.32 f | 113 ± 1.8 b,c | 4.2 ± 0.07 b,c |
| H23    | 3710                     | 2410                            | 3.3 ± 0.03 c,b | 18.2 ± 0.41 b,c | 78 ± 1.8 a | 4.1 ± 0.17 b |
| H24    | 3710                     | 2890                            | 3.3 ± 0.05 b,c | 16.7 ± 0.29 a | 75.1 ± 1.8 a | 4.3 ± 0.12 c,d |

* The values designated by the different small letters (a, b, c, d, ... ) are significantly different (α = 0.05).
In general, the higher the speed of both the disc and classifier, the lower the values of \(d_{10}\), \(d_{50}\), and \(d_{90}\). The results of the variance analysis of the parameters that characterize the particle size of the micronized OH are presented in Table 3. The speed of rotation of both the disc \((v_d)\) and classifier \((v_k)\) had a significant influence on the parameters characterizing the PSD of the OH. However, according to the values obtained by the \(F\)-test, the speed of the classifier had the highest influence on \(d_{10}\), \(d_{50}\), and \(d_{90}\), whereas the speed of the rotor disc had a crucial effect on the parameter Span.

Table 3. The analysis of variance for the parameters characterizing the PSD of pulverized oat husk.

| Parameter | Source of Variance | Sum of Squares | Degrees of Freedom | Mean Square | \(F\)-Test | \(p\)-Value |
|-----------|-------------------|----------------|--------------------|-------------|------------|------------|
| \(d_{10}\) | \(v_d\)* | 5.34 | 3 | 1.78 | 283 | 0.0001 |
| \(v_k\) | 27.06 | 5 | 5.41 | 859 | 0.0001 |
| \(v_d \cdot v_k\) | 17.43 | 15 | 1.16 | 184 | 0.0001 |
| Error | 0.756 | 120 | 0.006 | - | - |
| \(d_{50}\) | \(v_d\)* | 943.5 | 3 | 314.5 | 692.4 | 0.0001 |
| \(v_k\) | 4976.3 | 5 | 995.3 | 2191.3 | 0.0001 |
| \(v_d \cdot v_k\) | 3592.5 | 15 | 239.5 | 527.3 | 0.0001 |
| Error | 54.5 | 120 | 0.5 | - | - |
| \(d_{90}\) | \(v_d\)* | 23.225 | 3 | 7.742 | 647.4 | 0.0001 |
| \(v_k\) | 147.444 | 5 | 29.489 | 2466 | 0.0001 |
| \(v_d \cdot v_k\) | 94.26 | 15 | 6.284 | 525.5 | 0.0001 |
| Error | 1435 | 120 | 12 | - | - |
| Span | \(v_d\)* | 11.3 | 5 | 2.26 | 260.5 | 0.0001 |
| \(v_k\) | 27.4 | 15 | 1.83 | 211.6 | 0.0001 |
| Error | 1.04 | 120 | 0.009 | - | - |

* \(v_d\)—speed of the disc, \(v_k\)—speed of the classifier.

Moreover, in the case of all of the parameters, significant interactions were found between the speed of the disc and the classifier. The changes in these parameters were described using multiple linear regression:

\[
Y = a_1 \times v_d + a_2 \times v_k + b \pm e \tag{4}
\]

where \(Y\) is the dependent variable, \(a_1\) and \(a_2\) are the regression coefficients, \(b\) is the intercept, \(v_d\) and \(v_k\) are the speeds of the disc and classifier, respectively, and \(e\) is the model’s error.

The results of the regression are summarized in Table 4. All of the parameters describing the PSD showed significant relations, and the coefficient of determination ranged from 0.722 (\(d_{10}\)) to 0.785 (\(d_{50}\)). This shows that the proposed equations generally described the changes in \(d_{10}\), \(d_{50}\), and \(d_{90}\) well with respect to the function of the rotation speed of the disc and the classifier. In the case of Span, the lowest \(R^2\) values were found (0.577). However, for both the coefficient of determination and the equation parameters, \(p\)-values were statistically significant.

3.3. Antioxidant Activity (AA) of OH

OH is rich in phenolic acids such as ferulic acid, \(p\)-coumaric acid, and vanillic acid, which have an influence on its AA [50]. Vagra et al. [51] studied the phenolic composition and AA of oat hull and groats using 20 oat genotypes and found AA that was several times higher in husks compared to groats. Lower values of \(d_{10}\), \(d_{50}\), and \(d_{90}\) resulted in a lower EC\(_{50}\) index in the case of all AA assays and consequently higher antioxidant activity (Table 5). The values of EC\(_{50}\) for CHEL changed from 27.67 mg dm/mL (H24) to 37.05 mg dm/mL (H1). The values of EC\(_{50}\) for ABTS ranged from 35.3 mg dm/mL (H23) to 44.9 mg dm/mL (H6), and the values for DPPH ranged from 76.9 mg dm/mL (H11) to 85.1 mg dm/mL (H7).
Table 4. Results of equations describing the relationships between the working parameters of the mill and the parameters describing the PSD of micronized OH.

| Dependent Variable | Equation Parameter | Standardized Coefficient of Regression | Standard Error | Coefficient of Regression | Standard Error | t (213) | p-Value | R² | Model’s Error |
|--------------------|--------------------|----------------------------------------|----------------|----------------------------|----------------|---------|---------|-----|--------------|
| d₁₀                | Intercept          | -0.846                                 | 0.0361         | -0.00144                   | 0.0006         | 20.262  | 0.00001 | 0.722 | 0.740        |
| v₁                | v₁                 | -0.077                                 | 0.0361         | -0.00026                   | 0.00012        | -2.118  | 0.03534 |     |              |
| d₅₀                | Intercept          | -0.822                                 | 0.038          | -0.00579                   | 0.00073        | 21.718  | 0.00001 | 0.785 | 8.79         |
| v₅₀               | v₅₀                | -0.139                                 | 0.038          | -0.00532                   | 0.00148        | -3.677  | 0.00001 |     |              |
| d₉₀                | Intercept          | -0.716                                 | 0.03178        | -0.00587                   | 0.000261       | 22.52   | 0.00001 | 0.784 | 31.5         |
| v₉₀               | v₉₀                | -0.522                                 | 0.03178        | -0.00852                   | 0.00519        | -16.42  | 0.00001 |     |              |
| Span              | Intercept          | -0.6143                                | 0.0445         | -0.00016                   | 0.000012       | -13.79  | 0.00001 | 0.577 | 0.715        |
|                   | v₆₄                | 0.4471                                 | 0.0445         | 0.00006                    | 0.00006        | 10.02   | 0.00001 |     |              |

* v₆₄—speed of the disc, v₅₀—speed of the classifier.

Table 5. Antioxidant activity of OH.

| Sample | EC₅₀CHEL [mg dm/mL] | EC₅₀ABTS [mg dm/mL] | EC₅₀DPPH [mg/mL] |
|--------|---------------------|---------------------|------------------|
| H1     | 37.05 ± 1.35        | 41.3 ± 1.2          | 80.5 ± 1.2       |
| H2     | 37.79 ± 0.40        | 42.2 ± 2.1          | 82.5 ± 1.2       |
| H3     | 36.23 ± 0.36        | 43.2 ± 2.1          | 80.9 ± 1.2       |
| H4     | 36.40 ± 0.41        | 44.9 ± 1.8          | 82.2 ± 2.4       |
| H5     | 35.27 ± 0.34        | 44.1 ± 1.4          | 82.1 ± 1.7       |
| H6     | 28.20 ± 0.36        | 41.9 ± 1.6          | 77.8 ± 1.7       |
| H7     | 36.30 ± 0.45        | 44.4 ± 2.0          | 85.1 ± 1.4       |
| H8     | 34.47 ± 0.64        | 43.0 ± 2.0          | 81.3 ± 1.1       |
| H9     | 34.83 ± 0.57        | 40.4 ± 1.3          | 82.1 ± 1.7       |
| H10    | 29.75 ± 0.85        | 37.9 ± 2.3          | 77.5 ± 1.2       |
| H11    | 29.73 ± 0.55        | 38.1 ± 1.6          | 76.9 ± 1.3       |
| H12    | 28.06 ± 0.49        | 35.9 ± 1.6          | 78.7 ± 3.4       |
| H13    | 35.64 ± 0.82        | 42.6 ± 2.6          | 81.8 ± 3.7       |
| H14    | 30.51 ± 0.36        | 41.2 ± 2.7          | 79.4 ± 2.7       |
| H15    | 33.27 ± 1.11        | 42.9 ± 2.5          | 83.2 ± 0.52      |
| H16    | 32.67 ± 0.82        | 39.9 ± 2.1          | 81.0 ± 3.8       |
| H17    | 30.6 ± 1.09         | 36.4 ± 2.0          | 77.2 ± 1.8       |
| H18    | 28.77 ± 0.79        | 36.5 ± 0.9          | 78.0 ± 1.2       |
| H19    | 36.37 ± 0.91        | 41.4 ± 1.67         | 83.6 ± 1.02      |
| H20    | 37.33 ± 1.11        | 42.4 ± 2.2          | 83.0 ± 2.1       |
| H21    | 34.60 ± 0.72        | 37.5 ± 2.1          | 80.9 ± 1.7       |
| H22    | 31.33 ± 1.10        | 37.2 ± 2.1          | 77.1 ± 1.6       |
| H23    | 31.27 ± 0.76        | 35.3 ± 2.1          | 77.9 ± 2.2       |
| H24    | 27.67 ± 2.59        | 35.6 ± 1.6          | 79.7 ± 3.2       |

The values designated by the different small letters (a, b, c, d, . . .) are significantly different (α = 0.05).

The coefficients of correlation between d₁₀, d₅₀, d₉₀ and CHEL were statistically significant and were included in Table 6. A similar relation was found between the particle size of micronized wheat bran and antioxidant activity in the study by Rosa et al. [52], in which a 1.5-fold increase was observed in the antioxidant capacity of bran when d₅₀ was decreased from 172 to 30 μm. Additionally, other authors have observed that antioxidant activity increased as a result of the micronization of different fibre-rich plant materials [53–55]. This fact can be explained by the expansion of the surface area of particles and the destruction of the dietary fibre matrix, which resulted in the release of some antioxidative compounds from the matrix. Thus, the ultra-fine grinding of fibre-rich plant materials could be beneficial in enhancing the antioxidant activity and the pro-health value of food. In addition, the reduction of particle size by micronization increases the bioavailability of many compounds, and pulverized material is easily absorbed by the human body [18]. The relatively lower values of the antioxidant activity
of OH can result from the interactions of polyphenols with carbohydrates originating from the cell wall such as cellulose or dietary fibres [56]. However, the polyphenols from such complexes could be released in the colon when exposed to various enzymes and microorganisms [57–60].

Table 6. Correlation coefficients and $p$-values describing the relation between the parameters of PSD and antioxidant activity of OH.

| Parameter | CHEL * | ABTS | DPPH |
|-----------|--------|------|------|
|           | $r$   | $p$-Value | $r$   | $p$-Value | $r$   | $p$-Value |
| $d_{10}$  | 0.822 | $p = 0.0001$ | 0.752 | $p = 0.0001$ | 0.854 | $p = 0.0001$ |
| $d_{50}$  | 0.887 | $p = 0.0001$ | 0.696 | $p = 0.0001$ | 0.795 | $p = 0.0001$ |
| $d_{90}$  | 0.845 | $p = 0.0001$ | 0.908 | $p = 0.0001$ | 0.798 | $p = 0.0001$ |

* CHEL—chelating power, ABTS—radical scavenging activity, DPPH—antiradical activity.

3.4. Phenolic Acids Profile

The UPLC-MS/MS analysis detected seven phenolic acids in the OH samples. The three samples of OH (H12, H17 and H24) with the highest degree of fineness (the lowest values of $d_{50}$) were not statically different according to phenolic acid profile. The dominant phenolic acid was ferulic acid (on average 454.56 µg/mg d.m.). This compound represented about 95% of all of the detected phenolic acids. The following phenolic acids were detected in the OH samples: caffeic, $p$-hydroxybenzoic, vanilic, syringic, and synapic acid (Table 7). This compound has a lot of applications. It is an ingredient in many drugs, functional foods, and nutraceuticals [61]. Ferulic acid (4-hydroxy-3-methoxycinnamic acid) shows anticancer, anti-inflammatory, antimicrobial, antithrombotic, and hepatoprotective activity [62]. Although ferulic acid is the main phenolic acid of OH, it is not effortlessly available from this material because it is covalently linked with lignin and other biopolymers [62]. However, the enzymatic production of ferulic acid from lignocellulosic materials is possible [63,64].

Table 7. Total phenolic acids profile (free and bound, µg/mg d.m.) in OH.

| Phenolic Acid         | Sample |     |     |     |
|-----------------------|--------|-----|-----|-----|
|                       | H12    | H17 | H24 |     |
| Protocatechuic acid   | 0.73 ± 0.02 $^a$ | 0.76 ± 0.01 $^a$ | 0.74 ± 0.05 $^a$ |     |
| $p$-Hydroxybenzoic    | 5.12 ± 0.07 $^a$ | 5.13 ± 0.17 $^a$ | 5.05 ± 0.16 $^a$ |     |
| Vanillic              | 4.26 ± 0.31 $^a$ | 4.24 ± 0.27 $^a$ | 4.33 ± 0.12 $^a$ |     |
| Caffeic               | 6.26 ± 0.01 $^a$ | 6.28 ± 0.10 $^a$ | 6.18 ± 0.21 $^a$ |     |
| Syringic              | 3.14 ± 0.17 $^a$ | 3.07 ± 0.16 $^a$ | 3.03 ± 0.18 $^a$ |     |
| $p$-Coumaric          | Nq ** | Nq  | Nq  |     |
| Ferulic               | 438.19 ± 14.32 $^a$ | 427.93 ± 8.18 $^a$ | 433.37 ± 6.79 $^a$ |     |
| Synapic               | 1.84 ± 0.07 $^a$ | 1.92 ± 0.13 $^a$ | 1.83 ± 0.11 $^a$ |     |
| Salycilic             | Nd     | Nd  | Nd  |     |
| Total                 | 459.55 ± 14.22 $^a$ | 449.32 ± 8.28 $^a$ | 454.52 ± 7.22 $^a$ |     |

The values designated by the same small letters ($a$) are not significantly different ($\alpha = 0.05$). ** Nq—above limit of detection and below limit of quantification, Nd—below limit of detection.

4. Conclusions

This study demonstrated that the sterilization of OH decreases the moisture content, thereby resulting in a very dry material with appropriate microbiological purity. The proposed method of micronization effectively reduced the particle size of the sterilized husk. In addition, PSD can be modified by changing the working parameters of the mill that is used. By increasing the speed of hammers and classifier, ground OH with a median particle size below 20 µm can be obtained. Moreover, the association between the working parameters of the mill and the parameters describing the PSD of OH can be described by using a multilinear equation with respect to the speeds of both the disc and the classifier. Most importantly, the obtained material is rich in phenolic compounds, and a
positive correlation was found between the size of the particles and the antioxidant activity. The UPLC-MS/MS analysis showed that ferulic acid represented about 95% of all of the detected phenolic acids. Therefore, the powder obtained from the OH can be used as a functional food additive and as a source of ferulic acid for industrial purposes.

**Author Contributions:** Conceptualization, D.D., W.T. and U.G.-D.; methodology, D.D., W.T. and U.G.-D.; validation, D.D., formal analysis, D.D.; investigation, D.D., W.T. and U.G.-D.; data curation, D.D.; writing—original draft preparation, D.D.; writing—review and editing, D.D.; supervision, D.D. All authors have read and agreed to the published version of the manuscript.

**Funding:** The research was partially founded by the project “Obtaining a fiber preparation from the husk and fruit-seed coat of oat grain with the use of innovative hulling and crushing technologies” (Grant Number POIR.01.01.01.-00-0289/17) co-financed by the European Union from the European Regional Development Fund under the Intelligent Development Operational Programme for 2014–2020.

**Institutional Review Board Statement:** Not applicable.

**Informed Consent Statement:** Not applicable.

**Data Availability Statement:** The data presented in this study are available upon request from the corresponding author.

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

**References**

1. Shewry, P.R.; Piironen, V.; Lampi, A.M.; Nyström, L.; Li, L.; Rakszegi, M.; Fraš, A.; Boros, D.; Gebruers, K.; Courtin, C.M.; et al. Phytochemical and fiber components in oat varieties in the healthgrain diversity screen. *J. Agric. Food Chem.* **2008**, *56*, 9777–9784. [CrossRef] [PubMed]

2. Nurullaeva, D.; Farmanaova, N.; Luzhanin, V.; Povidysh, M.; Orlova-Lebedkova, A. Chemical components and biological activity of oats seed-avena sativa I. *Int. J. Pharm. Res.* **2020**, *12*, 221–229. [CrossRef]

3. Demirel, F.; Germec, M.; Coban, H.B.; Turhan, I. Optimization of dilute acid pretreatment of barley husk and oat husk and determination of their chemical composition. *Cellulose* **2018**, *25*, 6377–6393. [CrossRef]

4. McDonald, P.; Edwards, R.; Greenhalgh, J.F.D.; Morgan, C.; Sinclair, L.; Wilkinson, R.G. Grass and forage crops. In *Oats: Chemistry and Technology*, 2nd ed.; Pearson: Harlow, UK, 2011; pp. 553–554.

5. Emmons, C.L.; Peterson, D.M. Antioxidant activity and phenolic contents of oat groats and hulls. *Cereal Chem.* **1999**, *76*, 902–906. [CrossRef]

6. Piątkowska, E.; Witkowicz, R.; Pisulewska, E. Antioxidant properties of selected cultivars of oats. *Food Sci. Technol. Qual.* **2010**, *14*, 100–107. [CrossRef]

7. Marlett, J.A.; McBurney, M.I.; Slavin, J.L. Position of the American Dietetic Association: Health implications of dietary fiber. *J. Am. Diet. Assoc.* **2002**, *102*, 993–1000. [CrossRef]

8. De Oliveira, J.P.; Bruni, G.P.; Lima, K.O.; El Halal, S.L.M.; DA Rosa, G.S.; Dias, A.R.G.; Zavareze, E.D.R. Cellulose fibers extracted from rice and oat husks and their application in hydrogel. *Food Chem.* **2017**, *221*, 153–160. [CrossRef]

9. Welch, R.W.; Hayward, M.V.; Jones, D.I.H. The composition of oat husk and its variation due to genetic and other factors. *J. Sci. Food Agric.* **1983**, *34*, 417–426. [CrossRef]

10. Girardet, N.; Webster, F.H. Oat milling: Specifications, storage, and processing. In *Oats: Chemistry and Technology*, 2nd ed.; Webster, F.H., Ed.; Elsevier: Amsteldam, The Netherlands, 2011; pp. 301–319.

11. Piwitska, M.; Wywisz, J.; Wierzbicka, A. Effect of micronization of high-fiber oat powder and vacuum-drying on pasta quality. *CYTA J. Food* **2016**, *14*, 433–439. [CrossRef]

12. Lapčíková, B.; Burešová, I.; Lapčík, L.; Dabash, V.; Valenta, T. Impact of particle size on wheat dough and bread characteristics. *Food Chem.* **2019**, *297*, 124938. [CrossRef]

13. Kurek, M.A.; Sokolova, N. Optimization of bread quality with quinoa flour of different particle size and degree of wheat flour replacement. *Food Sci. Technol.* **2020**, *40*, 307–314. [CrossRef]

14. Drakos, A.; Kyriakakis, G.; Evageliou, V.; Protonotariou, S.; Mandala, I.; Ritzoulis, C. Influence of jet milling and particle size on the composition, physicochemical and mechanical properties of barley and rye flours. *Food Chem.* **2017**, *215*, 326–332. [CrossRef]

15. Frohlich, P.; Young, G.; Bourné, L.; Borsuk, Y.; Sarkar, A.; Sopiwnyk, E.; Pickard, M.; Dyek, A.; Malcolmson, L. Effect of premilling treatments on the functional and bread-baking properties of whole yellow pea flour using micronization and pregermination. *Cereal Chem.* **2019**, *96*, 895–907. [CrossRef]

16. Xue, X.; Wang, J.; Li, S.; Zhang, X.; Dong, J.; Gui, L.; Chang, Q. Effect of micronised oat bran by ultrafine grinding on dietary fibre, texture and rheological characteristic of soft cheese. *Int. J. Food Sci. Technol.* **2020**, *55*, 578–588. [CrossRef]
17. Protonotariou, S.; Drakos, A.; Evageliou, V.; Ritzoulis, C.; Mandala, I. Sieving fractionation and jet mill micronization affect the functional properties of wheat flour. *J. Food Eng.* 2014, 134, 24–29. [CrossRef]

18. Chen, T.; Zhang, M.; Bhandari, B.; Yang, Z. Micronization and nanosizing of particles for an enhanced quality of food: A review. *Crit. Rev. Food Sci. Nutr.* 2018, 58, 993–1001. [CrossRef]

19. Zhu, F.M.; Du, B.; Li, J. Effect of ultrafine grinding on physicochemical and antioxidant properties of dietary fiber from wine grape pomace. *Food Sci. Technol. Int.* 2014, 20, 55–62. [CrossRef]

20. Hussain, S.; Li, J.; Jin, W.; Yan, S.; Wang, Q. Effect of micronisation on dietary fibre content and hydration properties of lotus node powder fractions. *Int. J. Food Sci. Technol.* 2018, 53, 590–598. [CrossRef]

21. Speroni, C.S.; Guerra, D.R.; Bender, A.B.B.; Stiebe, J.; Ballus, C.A.; da Silva, L.P.; Lozano-Sánchez, J.; Emanuelli, T. Micronization increases the bioaccessibility of polyphenols from granulo metrically separated olive pomace fractions. *Food Chem.* 2020, 344, 128689. [CrossRef] [PubMed]

22. Bender, A.B.B.; Speroni, C.S.; Moro, K.I.B.; Morisso, F.D.P.; dos Santos, D.R.; da Silva, L.P.; Penna, N.G. Effects of micronization on dietary fiber composition, physicochemical properties, phenolic compounds, and antioxidant capacity of grape pomace and its dietary fiber concentrate. *LWT Food Sci. Technol.* 2020, 117, 108652. [CrossRef]

23. Dziki, D.; Laskowski, J.; Przyypek-Ochab, D. Energy consumption at grinding of black oat grain. *Agric. Eng.* 2020, 24, 45–54. [CrossRef]

24. AOAC. *Official Methods of Analysis of AOAC International*, 18th ed.; Horwitz, W., Latimer, G.W., Jr., Eds.; Revision 4; AOAC International: Gaithersburg, MA, USA, 2010.

25. ISO. 21527-1-2:2008—Microbiology of Food and Animal Feeding Stuffs—Horizontal Method for the Enumeration of Yeasts and Molds, 1st ed.; International Standardization Organization: Geneva, Switzerland, 2008.

26. ISO. 4833-1 Microbiology of the Food Chain—Horizontal Method for the Detection and Enumeration of Microorganisms—Part 1: Colony count at 30 °C by the Pour Plate Technique; ISO: Geneva, Switzerland, 2013.

27. ISO. 7251 Microbiology of Food and Animal Feeding Stuffs—Horizontal Method for the Detection and Enumeration of Prescott Escherichia Coli—Most Probable Number Technique; ISO: Geneva, Switzerland, 2005.

28. ISO. 6579 Microbiology of the Food Chain—Horizontal Method for the Detection, Enumeration and Serotyping Of Salmonella—Part 1: Detection Of Salmonella spp.; ISO: Geneva, Switzerland, 2017.

29. PN-EN ISO. 7932:2005—Food and Feed Microbiology—Horizontal Method for the Enumeration of Presumptive Bacillus Cereus—Colonies Enumeration Method At 30 °C; ISO: Geneva, Switzerland, 2005.

30. Dziki, D.; Tarasiuk, W.; Lysiak, G.; Jochymek, P. The study of particle size distribution of micronized oat bran layer. *Agric. Eng.* 2020, 24, 45–54. [CrossRef]

31. Ma, S.; Wang, C.; Li, L.; Wang, X. Effects of particle size on the quality attributes of wheat flour made by the milling process. *Cereal Chem.* 2020, 97, 172–182. [CrossRef]

32. Liu, T.Y.; Ma, Y.; Yu, S.F.; Shi, J.; Xue, S. The effect of ball milling treatment on structure and porosity of maize starch granule. *Innov. Food Sci. Emerg. Technol.* 2011, 12, 586–593. [CrossRef]

33. Złotek, U.; Kasań, M.; Gawkil-Dziki, U.; Szymanowska, U.; Baraniak, B.; Jakuczyk, A. Antioxidant activity of the aqueous and methanolic extracts of coffee beans (*Coffea Arabica L.*). *Acta Sci. Technol. Aliment.* 2016, 15, 281–288. [CrossRef]

34. Guo, J.T.; Lee, H.L.; Chiang, S.H.; Lin, F.I.; Chang, C.Y. Antioxidant properties of the extracts from different parts of broccoli in Taiwan. *J. Food Drug Anal.* 2001, 9, 96–101. [CrossRef]

35. Gawkil-Dziki, U.; Świeca, M.; Dziki, D.; Baraniak, B.; Tomilo, J.; Czyzy, J. Quality and antioxidant properties of broths enriched with dry onion (*Allium cepa L.*) skin. *Food Chem.* 2013, 138, 1621–1628. [CrossRef]

36. Brand-Williams, W.; Cuvelier, M.E.; Berset, C. Use of a free radical method to evaluate antioxidants activity. *LWT Food Sci. Technol.* 1995, 28, 25–30. [CrossRef]

37. Re, R.; Pellegrini, N.; Proteggente, A.; Pannala, A.; Yang, M.; Rice-Evans, C. Antioxidant activity applying an improved ABTS radical cation decolorization assay. *Free Radic. Biol. Med.* 1999, 26, 1231–1237. [CrossRef]

38. Romanikiewicz, D.; Hassoon, W.H.; Cacak-Pietrzak, G.; Sobczyk, M.; Wirkowska-WojdyBa, M.; CegliNska, A.; Dziki, D. The effect of chia seeds (*Salvia hispanica L.*) addition on quality and nutritional value of wheat bread. *J. Food Qual.* 2017, 2017, 7352631. [CrossRef]

39. Plavsic, D.; Skrinjar, M.; Psodorov, D.; Saric, L.; Psodorov, D.; Varga, A.; Mandic, A. Mycopopulations of grain and flour of wheat, corn and buckwheat. *Food Feed Res.* 2017, 44, 39–45. [CrossRef]

40. Cardoso, R.V.C.; Fernandes, Â.; Heleno, S.A.; Rodrigues, P.; González-Paramás, A.M.; Barros, L.; Ferreira, I.C.F.R. Physicochemical characterization and microbiology of wheat and rye flours. *Food Chem.* 2019, 280, 123–129. [CrossRef]

41. Aydin, A.; Peter, P.; Smulders, F.J.M. The physico-chemical and microbiological properties of wheat flour in Thrace. *Turk. J. Agric. For.* 2009, 33, 445–454. [CrossRef]

42. Wroniak, M.; Chlebowska-Smigiel, A. Impact of purity of rapeseed and oil purification method on selected properties of cold-pressed oils. *Żywność Nauk. Technol. Jakości* 2013, 4, 133–139. [CrossRef]

43. Dziki, D. The crushing of wheat kernels and its consequence on the grinding process. *Powder Technol.* 2008, 185, 181–186. [CrossRef]

44. Jung, H.; Lee, Y.J.; Yoon, W.B. Effect of moisture content on the grinding process and powder properties in food: A review. *Processes* 2018, 6, 69. [CrossRef]
45. Kurek, M.; Wyrwisz, J.; Piwińska, M.; Wierzbicka, A. The effect of oat fibre powder particle size on the physical properties of wheat bread rolls. *Food Technol. Biotechnol.* 2016, 54, 45–51. [CrossRef] [PubMed]

46. Piwińska, M.; Wyrwisz, J.; Kurek, M.; Wierzbicka, A. Effect of oat β-glucan fiber powder and vacuum-drying on cooking quality and physical properties of pasta. *CYTA J. Food* 2016, 14, 101–108. [CrossRef]

47. Song, D.; Wang, Z.; Zhao, Z.; Jing, X.; Zhang, L. Effects of Pretreatment Methods of Oat on Textural and Sensory Properties of Harbin Sausage. *J. Chin. Inst. Food Sci. Technol.* 2017, 17, 82–88. [CrossRef]

48. Wang, L.; Flores, R.A. Effects of flour particle size on the textural properties of flour tortillas. *J. Cereal Sci.* 2000, 31, 263–272. [CrossRef]

49. Krishnakumar, I.M.; Ravi, A.; Kumar, D.; Kuttan, R.; Maliakel, B. An enhanced bioavailable formulation of curcumin using fenugreek-derived soluble dietary fibre. *J. Funct. Food* 2012, 4, 348–357. [CrossRef]

50. Peterson, D.M. Oat antioxidants. *J. Cereal Sci.* 2001, 33, 115–129. [CrossRef]

51. Varga, M.; Jójárt, R.; Fónad, P.; Mihály, R.; Palágyi, A. Phenolic composition and antioxidant activity of colored oats. *Food Chem.* 2018, 268, 153–161. [CrossRef] [PubMed]

52. Rosa, N.N.; Barron, C.; Gaiani, C.; Dufour, C. Ultra-fine grinding increases the antioxidant capacity of wheat bran. *J. Cereal Sci.* 2013, 57, 84–90. [CrossRef]

53. Zhu, K.X.; Huang, S.; Peng, W.; Qian, H.F.; Zhou, H.M. Effect of ultrafine grinding on hydration and antioxidant properties of wheat bran dietary fiber. *Food Res. Int.* 2010, 43, 943–948. [CrossRef]

54. Zhang, M.; Wang, F.; Liu, R.; Tang, X.; Zhang, Q.; Zhang, Z. Effects of superfine grinding on physicochemical and antioxidant properties of Lycium barbarum polysaccharides. *LWT Food Sci. Technol.* 2014, 58, 594–601. [CrossRef]

55. Zhao, X.; Zhu, H.; Zhang, G.; Tang, W. Effect of superfine grinding on the physicochemical properties and antioxidant activity of red grape pomace powders. *Powder Technol.* 2015, 286, 838–844. [CrossRef]

56. Jakobek, L. Interactions of polyphenols with carbohydrates, lipids and proteins. *Food Chem.* 2015, 175, 556–567. [CrossRef]

57. MacDonald, R.S.; Wagner, K. Influence of dietary phytochemicals and microorganisms on colon cancer risk. *J. Agric. Food Chem.* 2012, 60, 6728–6735. [CrossRef] [PubMed]

58. Palafoux-Carlos, H.; Ayala-Zavala, J.F.; González-Aguilar, G.A. The role of dietary fiber in the bioaccessibility and bioavailability of fruit and vegetable antioxidants. *J. Food Sci.* 2011, 76, R6–R15. [CrossRef]

59. Saura-Calixto, F. Dietary fiber as a carrier of dietary antioxidants: An essential physiological function. *J. Agric. Food Chem.* 2011, 59, 43–49. [CrossRef]

60. Tuohy, K.M.; Conterno, L.; Gasperotti, M.; Viola, R. Up-regulating the human intestinal microbiome using whole plant foods, polyphenols, and/or fiber. *J. Agric. Food Chem.* 2012, 60, 8776–8782. [CrossRef] [PubMed]

61. Kumar, N.; Pruthi, V. Potential applications of ferulic acid from natural sources. *Biotechnol. Rep.* 2012, 4, 86–93. [CrossRef]

62. Zhao, Z.; Moghadasian, M.H. Chemistry, natural sources, dietary intake and pharmacokinetic properties of ferulic acid: A review. *Food Chem.* 2008, 109, 691–702. [CrossRef] [PubMed]

63. Uraji, M.; Kimura, M.; Inoue, Y.; Kawakami, K.; Kumagai, Y.; Harazono, K.; Hatanaka, T. Enzymatic production of ferulic acid from defatted rice bran by using a combination of bacterial enzymes. *Appl. Biochem. Biotechnol.* 2013, 171, 1085–1093. [CrossRef] [PubMed]

64. Juhevica-Radenkova, K.; Kviesis, J.; Moreno, D.A.; Seglina, D.; Vallejo, F.; Valdovska, A.; Radenkovs, V. Highly-efficient release of ferulic acid from agro-industrial by-products via enzymatic hydrolysis with cellulose-degrading enzymes: Part I—The superiority of hydrolytic enzymes versus conventional hydrolysis. *Foods* 2021, 10, 782. [CrossRef]