


## Article

# Comparison and Characterization of the Structure and Physicochemical Properties of Three Citrus Fibers: Effect of Ball Milling Treatment

Zhanmei Jiang , Minghan Zhang, Yuxuan Huang, Chenglong Ma, Sinan Mu, Hongyu Li, Xianqi Liu, Yue Ma, Yue Liu and Juncai Hou \*

Key Laboratory of Dairy Science, Ministry of Education, College of Food Science, Northeast Agricultural University, Harbin 150030, China

\* Correspondence: jchou@neau.edu.cn; Tel.: +86-0451-13796820798

**Abstract:** Effects of ball milling (BM) on the structure and physicochemical properties of three types of citrus fibers were investigated. With the extension of the grinding time, the particle size of citrus fibers significantly decreased. Fourier transform infrared spectroscopy (FTIR) showed that the three citrus fibers had similar chemical groups, and more -OH and phenolic acid groups were exposed after BM, and pectin and lignin were not degraded. Scanning electron microscope (SEM) results showed that the appearance of particles changed from spherical to fragmented, irregular shapes. The water holding capacity (WHC), oil holding capacity (OHC), and water swelling capacity (WSC) of citrus fibers LM, JK, and FS reached the maximum value after BM of 2 h (increasing by 18.5%), 4 h (increasing by 46.1%), and 10 h (increasing by 38.3%), respectively. After 10 h BM, citrus fibers FS and JK had the highest adsorption capacity of cholesterol and sodium cholate, increasing by 48.3% and 48.6%, respectively. This indicates that BM transforms the spatial structure of citrus fibers and improves their physicochemical properties.

**Keywords:** citrus fiber; ball milling; functional properties; physicochemical properties



**Citation:** Jiang, Z.; Zhang, M.; Huang, Y.; Ma, C.; Mu, S.; Li, H.; Liu, X.; Ma, Y.; Liu, Y.; Hou, J. Comparison and Characterization of the Structure and Physicochemical Properties of Three Citrus Fibers: Effect of Ball Milling Treatment. *Foods* **2022**, *11*, 2665. <https://doi.org/10.3390/foods11172665>

Academic Editors: Nicole Roberta Giuggioli, Cristiana Peano and Giovanna Giacalone

Received: 19 July 2022

Accepted: 26 August 2022

Published: 1 September 2022

**Publisher's Note:** MDPI stays neutral with regard to jurisdictional claims in published maps and institutional affiliations.



**Copyright:** © 2022 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (<https://creativecommons.org/licenses/by/4.0/>).

## 1. Introduction

Citrus is one of the most cultivated fruit crops in the world, with annual production of more than 124 million tons [1]. Worldwide, the quantity of citrus by-products was estimated to exceed 1.5 million tons. The global citrus processing industry generates more than 110 million tons of citrus waste [2]. Citrus waste, including citrus peels, pulp residues, and seeds, accounts for 40–60% of the total fruit mass. In the process of citrus fruit processing, citrus waste is a burden to the environment without further treatment. In order to maximize the utilization of citrus waste, citrus pomace is widely applied in the pectin extraction industry [3].

Citrus fiber is the dietary fiber extracted from citrus waste. It consists mainly of pectin extracted from insoluble dietary fiber (IDF) [4]. Studies have shown that citrus fiber has higher total dietary fiber (TDF) and better physicochemical properties than cereal fiber, such as water holding capacity (WHC) and water swelling capacity (WSC). In addition, research has found that citrus fiber also has excellent physiological functional properties. Citrus fiber is composed of IDF, SDF, pectin, and fructooligosaccharides. These components selectively stimulate the growth and activity of the intestinal microbiota, especially lactobacillus and bifidobacterium. Therefore, the intake of dietary fiber has a positive impact on the development of gut microbes [5,6]. The water-soluble dietary fiber in citrus fiber can adsorb cholesterol and reduce cholesterol in the body [7]. The composition and content of dietary fiber are different among various kinds of citrus fibers, and are related to the source and extraction method of dietary fiber.

The physicochemical properties of citrus fiber have been studied extensively. Many hydroxyl groups are present in the cellulose side chain in citrus fiber, and can combine with H<sub>2</sub>O to generate more stable hydrogen bonds, leading to good WHC. Due to its loose surface structure and small space barrier, citrus fiber can contain more water and have WSC [8]. The adjacent cellulose chains form a stable fibrous structure through hydrogen bonds, which makes cellulose in citrus fiber have oil holding capacity (OHC) [9]. It is widely believed that dietary fiber (DF) can improve the function properties of foods; for example, incorporation of dietary fiber in some foods can improve the viscosity of food and the ability to form gels [8]. The authors of [10] added citrus fiber to ice cream and found that citrus fiber can improve the sensory properties and non-fusibility of ice cream. The authors of [11] added citrus fiber to gluten-free corn breads, which showed that citrus fiber can improve the WHC of bread, and the incorporation of citrus fiber reduced the firmness of bread. Gedikoğlu and Clarke reported that all ground beef meatballs with citrus fiber were lower in saturated fatty acids and higher in dietary fiber compared to the control samples [12]. The authors of [13] added citrus fiber to sausage and found it can also decrease residual nitrite levels.

A large amount of research has showed that the modification technology of citrus fiber can enhance the physicochemical properties of dietary fiber. The modification methods are divided into physical and chemical methods, of which the physical method is the most common application. It includes ultrasonic treatment, high pressure homogenization treatment, ultrafine grinding treatment, and extrusion treatment [14]. Among these, ball milling (BM) is a kind of physical modification method which has a low cost and is friendly to the environment [15].

BM treatment can effectively decrease the particle size, and change the surface area and physicochemical properties [16]. For instance, the authors of [17] showed that BM significantly increased the WHC of the insoluble fiber-rich fraction and cellulose. Furthermore, the authors of [18] found that rice bran dietary fiber can remarkably enhance WHC, WSC, and nitrite ion adsorption capacity after ultrafine grinding. The authors of [19] also reported that the surface of citrus dietary fiber was smoother and its fluidity was higher after ultrafine grinding, and the cation exchange ability and metal cation binding ability were improved.

Although BM treatment has been applied for modification of citrus dietary fiber, it has been not expounded whether there are remarkable differences in the characteristics of commercial citrus fibers of various sources after BM treatment. Thus, the main innovation of this study is to highlight the modification effect of ball milling on citrus fibers from different sources. A comparison and characterization of the structure and physicochemical properties of three commercial citrus fibers after ultrafine grinding treatment were undertaken in this study. Additionally, the characteristic differences of three commercial citrus fibers after BM treatment were compared and analyzed through detection of their soluble dietary fibers (SDFs), IDF and TDF content, particle size, WHC, OHC, WSC, cholesterol adsorption capacity (CAC), sodium cholate adsorption capacity, Fourier transform infrared spectroscopy (FTIR), and scanning electron microscopy (SEM). After comparing the structure and physicochemical properties of the three citrus fibers, we selected an optimal citrus fiber for further in-depth research. This investigation provides basic data support for modification of commercial citrus fibers and broadens their application in the food industry.

## 2. Materials and Methods

### 2.1. Materials

The citrus fiber FS (5.7% protein, 1.12% fat, 5.36% moisture, 3.75% ash) was acquired from Fiberstar Biotechnology Co., Ltd., River Falls, WI 54022, USA. The citrus fiber LM (5.91% moisture, 5.04% protein, 3.75% ash, 0.79% fat) was acquired from Laimeng Biotechnology Co., Ltd., Guangdong, China. The citrus fiber JK (5.94% moisture, 5.21% protein, 3.13% ash, 0.87% fat) was acquired from JinKangjun Biotechnology Co., Ltd., Guangdong,

China. Cholesterol, furfural, and sodium cholate was purchased from Solarbio Science and Technology, Ltd., Beijing, China. Soybean oil was purchased from Jiusan Grain and Oil Industry Group Co., Ltd., Heilongjiang, China.

### 2.2. Preparation of Modified Citrus Fiber by BM

Three citrus fiber powders were placed in the jar of a QM-ISP2 planetary ball mill (Nanjing University Instrument Factory, Nanjing, China). Mill balls (25 g) were added to the citrus fiber powders at the ratio of 1:8. The mill equipment was rotated at a speed of 560 rpm. BM was carried out for 2, 4, 6, 8, and 10 h. After BM, samples were stored for further use.

### 2.3. Determination of Citrus Fiber Components

Moisture was determined by drying a 10 g sample at  $130 \pm 3$  °C for 3 h (AOAC, 1997). Ash was produced at 550 °C for 2 h (AOAC, 2000). Protein was analyzed according to the Kjeldahl method, and the conversion factor was 6.25 (AOAC method 2001.11). Fat was calculated by weight loss after a 6-cycle extraction with petroleum ether (AOAC, 2000).

### 2.4. Determination of SDF, IDF and TDF Content

TDF, IDF, and SDF contents were measured by the AOAC method 991.43 (AOAC, 2000). TDF was determined as follows: 225 mL ethanol was added to the sample and precipitated for 1 h. Residue was washed with 78% ethanol, 95% ethanol, acetone, and dried at 105 °C. IDF and SDF were determined as follows: Enzyme digestate was filtered into residue and filtrate. Residue was washed and dried at 105 °C to obtain IDF. A quantity of 80 g filtrate and washing water was mixed with 320 mL ethanol and precipitated for 1 h. The residue was washed with 78% ethanol, 95% ethanol, and acetone, and dried at 105 °C to obtain SDF.

### 2.5. Particle Size Analysis

The particle size distribution and median particle size of three citrus fibers were measured according to the authors of [20] using a laser diffraction particle analyzer (HYL-1076, Haoyu Technology Co., Ltd., Liaoning, China). The sample groups and control groups were diluted with deionized water to a concentration of 0.5 mg/mL. Further, Ultrasonic-assisted dispersion was used before measurement. The refractive index of the sample was set at 1.460, and the absorption rate was set at 0.1.

### 2.6. Determination of WHC

WHC of the samples was analyzed according to the method described by [21] with a small modification. Initially, each sample (1.0000 g) was mixed with distilled water (20 mL) for 24 h, and the mixture was centrifuged ( $3000 \times g$ , 15 min). The calculation formula of WHC is as follows:

$$\text{WHC (g/g)} = \frac{M_2 - M_1}{M_0}$$

where  $M_2$  is the weight of the sample in the centrifuge tube after supernatant removal,  $M_1$  is the weight of the centrifuge tube, and  $M_0$  is the weight of the sample.

### 2.7. Determination of OHC

OHC was determined using a modified method from [22]. Each sample (0.5000 g) was mixed with 5 mL of oil and kept for 24 h, and then centrifuged ( $3000 \times g$ , 15 min).

The calculation formula of OHC is as follows:

$$\text{OHC (g/g)} = \frac{M_2 - M_1}{M_0}$$

where  $M_2$  is the weight of the centrifuge tube and oil-contained sample,  $M_1$  is the weight of the centrifuge tube, and  $M_0$  is the weight of the sample.

### 2.8. Determination of WSC

WSC was determined using a method described by [23]. Initially, 0.2 g of citrus fiber samples was weighted. Subsequently, distilled water (10 mL) was added to this vessel, and it was stored for 24 h at room temperature.

The calculation formula of WSC is as follows:

$$\text{WSC (mL/g)} = \frac{V_2 - V_1}{M_0}$$

where  $V_2$  is the volume of the hydrated sample,  $V_1$  is the volume of the dried sample, and  $M_0$  is the weight of the dried sample.

### 2.9. Determination of CAC

CAC was determined using a modified method [24].

Fresh egg yolk was diluted 10-fold with distilled water. Mixtures of samples (1.0 g) and diluted yolk (25 mL) at pH 2.0 and 7.0, respectively, were shaken at 37 °C (120 rpm, 2 h). Then, it was centrifuged (2000 rpm, 15 min). The supernatant (1 mL) was diluted with the quintupling volume of glacial acetic acid. The supernatant dilution (400 µL), phthaldehyde color reagent (1.5 mL), and sulfuric acid (1 mL) were mixed. The absorbance was recorded at 550 nm.

The standard curve was drawn with the cholesterol standard solution (0.025, 0.050, 0.075, 0.100, and 0.125 mg/mL) as the abscissa and the absorbance value as the ordinate (the regression equation was  $y = 0.0159x + 0.0028$ ,  $R^2 = 0.9959$ ). CAC was calculated using the standard curve.

### 2.10. Determination of Adsorption Capacity of Sodium Cholate

The adsorption capacity of sodium cholate was determined using a modified method [25].

Briefly, 1.00 g sample and 0.1 g sodium cholate were dissolved with 50 mL of 0.15 mol/mL NaCl (pH 7.0), and shaken (120 rpm, 2 h) at 37 °C. Subsequently, this mixed solution was centrifuged (2000 rpm, 15 min) and supernatant was collected. Further, three reagents of supernatant (1 mL), concentrated sulfuric acid with a concentration of 45% (6 mL), and furfural at a concentration of 0.3% (1 mL) were mixed well and placed in a water bath (65 °C, 30 min). After cooling, the absorbance was measured at 620 nm. The standard curve was drawn with sodium cholate (0.025, 0.050, 0.075, 0.100, and 0.125 mg/mL) as the abscissa and the absorbance value as the ordinate (the regression equation was  $y = 0.5431x + 0.0221$ ,  $R^2 = 0.9980$ ). The adsorption capacity of sodium cholate was calculated using the standard curve.

### 2.11. Fourier Transform Infrared Spectroscopy (FTIR)

FTIR was measured by a reported method [26]. The infrared spectrum was performed in a total reflection FTIR instrument (AVATA 360, Nicolet, Thermo Fisher Scientific, Madison, WI, USA). The dry sample powder was mixed with KBr (1:100,  $v/v$ ), and the spectra were obtained between 400 and 4000  $\text{cm}^{-1}$ .

### 2.12. Scanning Electron Microscopy (SEM)

The micrograph was observed according to a method described by [27] using a SEM (EVO 18, ZEISS, Oberkochen, Germany) at 5.0 KV. Each micrograph of samples was taken at 500–2000× magnification.

### 2.13. Statistical Analysis

Statistical analyses were performed using an analysis of variance (ANOVA) procedure of the SPSS 25.0. Significant differences ( $p < 0.05$ ) of means were determined by the Duncan test, and the figures were drawn with Origin 2019. All tests were carried out in triplicate and the results are presented as mean values  $\pm$  standard deviation.

### 3. Results and Discussion

#### 3.1. The Content of IDF, SDF and TDF

Effects of BM on IDF, SDF, and TDF contents of three citrus fibers are shown in Table 1. The TDF content of FS, JK, and LM was significantly different, and the contents of TDF and IDF in citrus fiber LM were the highest among the three citrus fiber products ( $p < 0.05$ ). In addition, citrus fiber FS has the highest SDF content. The authors of [28] investigated the dietary fiber composition of five types of pummelo and showed that Shatianyu had the highest TDF content. The authors of [29] also demonstrated that citrus fibers from chemical industry sources had higher lignin and cellulose contents, whereas citrus fibers from food industry sources had higher hemicellulose and pectin contents. However, the author of [24] reported the chemical composition of five citrus fibers, and found that there were no remarkable distinctions in the contents of TDF, SDF and IDF in five citrus fibers. The above research results show that composition of citrus fiber from different sources was different.

**Table 1.** Effects of BM on IDF, SDF, and TDF contents of three citrus fibers (g/100 g).

| Sample  | IDF                        | SDF                        | TDF                        | IDF: SDF |
|---------|----------------------------|----------------------------|----------------------------|----------|
| sFS-0h  | 46.42 ± 0.28 <sup>j</sup>  | 30.82 ± 0.18 <sup>c</sup>  | 77.24 ± 1.00 <sup>e</sup>  | 1.51     |
| sFS-2h  | 45.72 ± 0.49 <sup>jk</sup> | 30.91 ± 0.61 <sup>c</sup>  | 76.64 ± 0.83 <sup>e</sup>  | 1.48     |
| sFS-4h  | 44.88 ± 0.73 <sup>k</sup>  | 31.52 ± 0.37 <sup>bc</sup> | 76.40 ± 0.72 <sup>ef</sup> | 1.42     |
| sFS-6h  | 43.31 ± 0.92 <sup>l</sup>  | 31.93 ± 0.07 <sup>b</sup>  | 75.25 ± 0.99 <sup>fg</sup> | 1.36     |
| sFS-8h  | 42.35 ± 0.23 <sup>lm</sup> | 32.19 ± 0.34 <sup>ab</sup> | 74.55 ± 0.10 <sup>g</sup>  | 1.31     |
| sFS-10h | 41.31 ± 0.45 <sup>m</sup>  | 32.73 ± 0.61 <sup>a</sup>  | 74.05 ± 0.16 <sup>g</sup>  | 1.26     |
| sJK-0h  | 65.28 ± 0.25 <sup>cd</sup> | 18.99 ± 0.11 <sup>j</sup>  | 84.27 ± 0.33 <sup>c</sup>  | 3.44     |
| sJK-2h  | 62.86 ± 0.74 <sup>e</sup>  | 20.76 ± 0.61 <sup>hi</sup> | 83.63 ± 0.72 <sup>cd</sup> | 3.03     |
| sJK-4h  | 61.41 ± 1.13 <sup>f</sup>  | 21.70 ± 1.4 <sup>gh</sup>  | 83.10 ± 0.27 <sup>cd</sup> | 2.82     |
| sJK-6h  | 59.32 ± 0.33 <sup>g</sup>  | 23.63 ± 0.53 <sup>f</sup>  | 82.95 ± 0.19 <sup>cd</sup> | 2.51     |
| sJK-8h  | 57.78 ± 0.66 <sup>h</sup>  | 25.09 ± 0.30 <sup>e</sup>  | 82.87 ± 0.65 <sup>d</sup>  | 2.30     |
| sJK-10h | 56.17 ± 1.28 <sup>i</sup>  | 26.50 ± 0.38 <sup>d</sup>  | 82.67 ± 1.28 <sup>d</sup>  | 2.12     |
| sLM-0h  | 70.48 ± 0.56 <sup>a</sup>  | 17.97 ± 1.05 <sup>j</sup>  | 88.46 ± 0.73 <sup>a</sup>  | 3.92     |
| sLM-2h  | 69.61 ± 0.51 <sup>a</sup>  | 18.23 ± 0.97 <sup>j</sup>  | 87.85 ± 1.30 <sup>a</sup>  | 3.82     |
| sLM-4h  | 67.35 ± 0.94 <sup>b</sup>  | 20.08 ± 0.54 <sup>i</sup>  | 87.43 ± 1.46 <sup>a</sup>  | 3.35     |
| sLM-6h  | 66.45 ± 0.76 <sup>bc</sup> | 20.92 ± 0.07 <sup>hi</sup> | 87.38 ± 0.68 <sup>b</sup>  | 3.18     |
| sLM-8h  | 65.94 ± 1.05 <sup>c</sup>  | 21.04 ± 0.91 <sup>hi</sup> | 86.98 ± 0.13 <sup>b</sup>  | 3.13     |
| sLM-10h | 64.35 ± 0.47 <sup>d</sup>  | 22.41 ± 0.23 <sup>g</sup>  | 86.53 ± 0.35 <sup>b</sup>  | 2.87     |

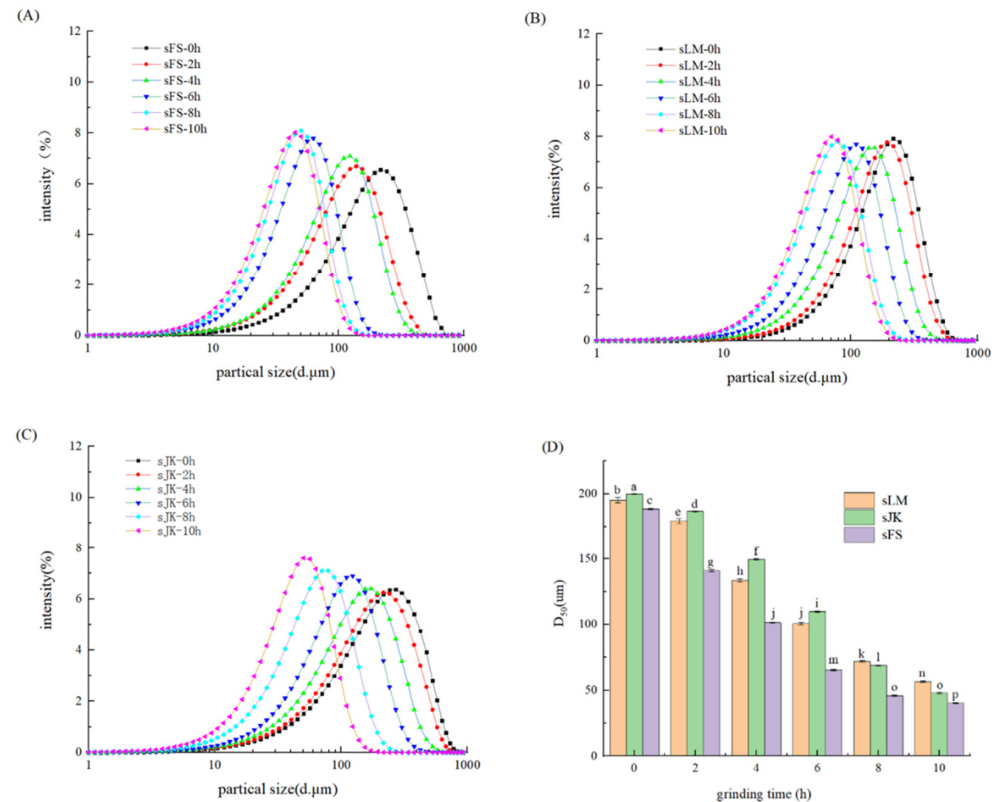
Note: Values with the different superscript letters in the same column are significantly different ( $p < 0.05$ ).

The contents of TDF and IDF of three citrus fibers were significantly decreased after BM, whereas the contents of SDF were significantly increased ( $p < 0.05$ ). Generally, the hydrogen bond between the molecules of IDF in citrus fiber was potentially broken during the process of BM, resulting in transfer of branch-chain celluloses into short-chain soluble amorphous celluloses [30]. Another possibility is that BM may destroy the cell structure of citrus pomace, thus releasing soluble polysaccharides in the cells, leading to the increase in SDF [31].

#### 3.2. Particle Size Distribution

Particle size analysis is an important indicator for evaluating physicochemical properties [32–34]. Effects of BM on particle size distribution and median particle size ( $D_{50}$ ) of three citrus fibers are shown in Figure 1A–D.  $D_{50}$  is the median particle size and is commonly used to represent the average particle size of powders [35]. From Figure 1A–C, the particle size distribution of three kinds of citrus fibers was moved to the left and became narrower in the grinding time between 2–10 h.  $D_{50}$  of FS, LM, and JK decreased from  $188.52 \pm 0.83 \mu\text{m}$ ,  $195.31 \pm 2.00 \mu\text{m}$ , and  $200.07 \pm 0.474 \mu\text{m}$  to  $40.44 \pm 0.31 \mu\text{m}$ ,  $56.65 \pm 0.50 \mu\text{m}$ , and  $47.85 \pm 0.42 \mu\text{m}$ , respectively. From Figure 1D, the particle size of JK

was the largest before modification, whereas the particle size of LM was the largest after 10 h of BM. In addition, it can be concluded that BM treatment has the greatest influence on the particle size of JK, which decreased by 152.22  $\mu\text{m}$ . This may be due to the different sources of three citrus fibers. The authors of [17] found that BM (5 and 10 h) remarkably ( $p < 0.05$ ) decreased the particle size of the carrot insoluble fiber-rich fraction to 58.4  $\mu\text{m}$  (−55.8%) and 12.4  $\mu\text{m}$  (−90.6%). A study by [36] showed that the  $D_{50}$  of citrus fibers gradually decreased with BM from 4 to 10 h under the same HPH pressure conditions ( $p < 0.05$ ).



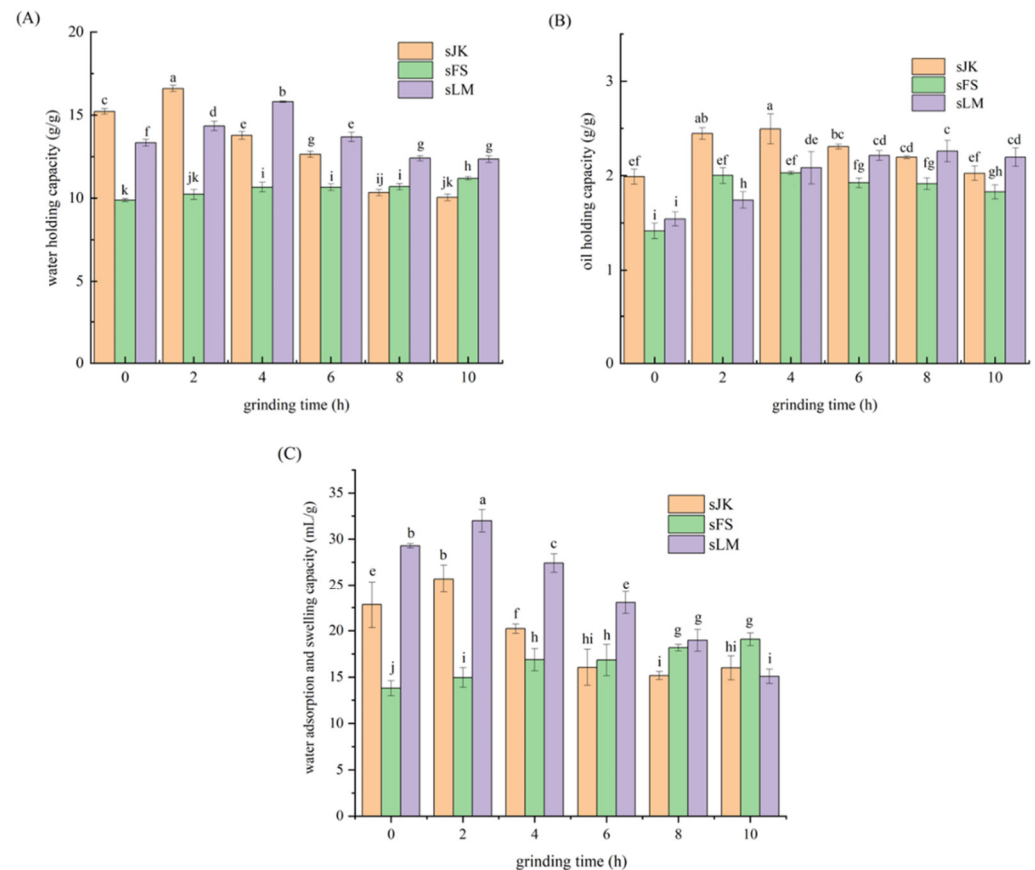
**Figure 1.** Effects of BM on particle size distribution of LM (A), JK (B), and FS (C). Average particle size of BM-treated citrus fibers (D). Error bars represent the standard deviation of the mean of triplicate experiments. Different letters (a–p) indicate significant difference ( $p < 0.05$ ).

Furthermore, Figure 1D also indicates that  $D_{50}$  of three dietary fibers greatly declined ( $p < 0.05$ ). This may be due to the grinding force generated by the impact between the balls during the grinding process, which makes the particle size of the three kinds of citrus fibers decrease continuously. Another possibility is that BM destroyed the cellular structure of citrus fibers and released small molecule polysaccharides, thus significantly reducing  $D_{50}$  of citrus fibers [37]. Consistent with our findings, BM treatment resulted in reduced particle sizes of potato, taro, and yam peels, and persimmon by-products [37]. According to [38], with the increase in grinding time,  $D_{50}$  of onion peel powder also decreased, but as the grinding time reached 18 h,  $D_{50}$  value did not decrease further.

### 3.3. Analysis of WHC

Effects of BM on WHC of three citrus fibers are shown in Figure 2A. WHC of three citrus fibers was  $15.21 \pm 0.17$  g/g,  $9.88 \pm 0.08$  g/g, and  $13.33 \pm 0.21$  g/g. After BM, WHC of citrus fibers JK and LM initially increased and then decreased. The reason for this may be that interfacial tension was reduced by BM and water binding sites were exposed to water [39]. During the BM process, the particle size of citrus fiber was reduced, and hydrophilic groups in the insoluble cellulose were exposed, thus resulting in the increase in WHC of LM and JK. The authors of [40] found that WHC of fibers was related to the porous structure formed by polysaccharide chains, which held a large amount of water

through hydrogen bonds; however, it could be damaged with longer milling times, thus reducing WHC of JK and LM. The authors of [41] also found that WHC of asparagus leaf by-product powder decreased gradually from 6.2% to 3.8% with increasing grinding time from 0 to 6 h ( $p < 0.05$ ).



**Figure 2.** Effect of BM on WHC (A), OHC (B), and WSC (C) of three citrus fibers (JK, FS, and LM). Error bars represent the standard deviation of the mean of triplicate experiments. Different letters (a–k) indicate significant differences ( $p < 0.05$ ).

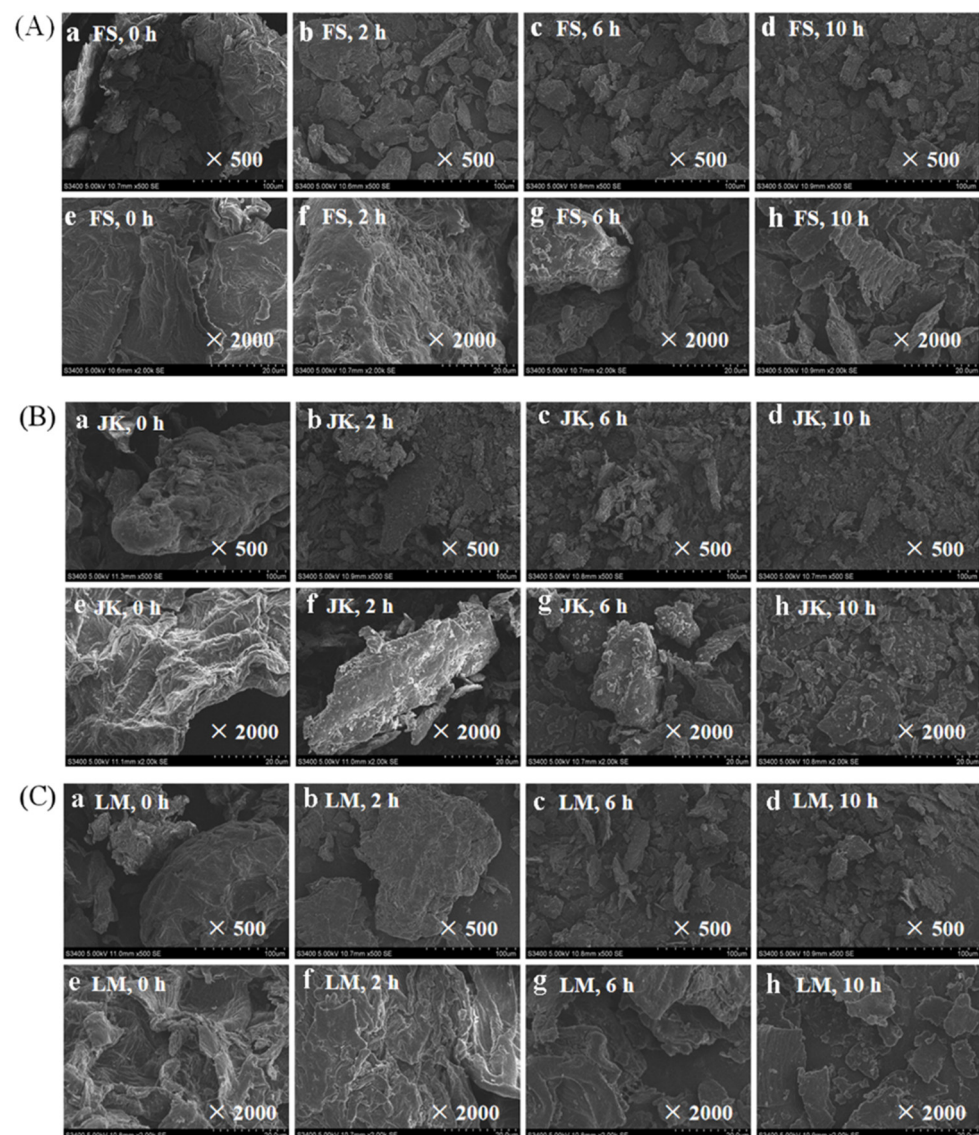
However, WHC of FS increased significantly with the increase in grinding time. Potentially, WHC of dietary fiber was positively correlated with the content of SDF [42]. Among the three citrus fibers, FS had the highest SDF content, and it gradually increased with the extension of grinding time, so WHC of FS also showed an increasing trend. Specifically, WHC of citrus fibers LM, JK, and FS was increased by 18.52%, 9.07%, and 13.15% ( $p < 0.05$ ), respectively, after being crushed by BM for 2, 4, and 10 h. LM and JK have more IDF (shown in Table 1).

### 3.4. Analysis of OHC

Citrus fiber has a large number of lipophilic groups, and the spatial structure is a loose network structure, which can absorb and bind a certain amount of oil, so it has OHC. Effects of BM on oil holding properties of three citrus fibers are shown in Figure 2B.

The authors of [23] showed that micronization improved OHC in Fraction 1 (>2 mm) of olive pomace, which was separated in a 2 mm sieve, but that of its Fraction 2 (<2 mm) was slightly declined ( $p < 0.05$ ). The authors of [43] found that there were no significant differences in OHC of four citrus fibers, indicating that homogenization had little effect on OHC. The authors of [3] modified citrus fibers by water media BM; the results showed that this modification method could further enhance OHC of citrus fibers.

In our study, OHC of three citrus fibers LM, JK and FS was significantly improved during BM ( $p < 0.05$ ). Compared with the untreated sample, OHC of LM and JK after BM for 4h was increased by 25.1% and 46.1%, and OHC of FS after BM for 8h increased by 41.8% ( $p < 0.05$ ). This is because the spatial structure of three citrus fibers was looser after BM, and more lipophilic groups were released. The morphology analysis (shown in Figure 3) was consistent with the change in OHC, indicating that a coarse surface, loose structure, and particles with good dispersion were more favorable to retain oils in citrus fibers [3]. However, after prolonged BM treatment, the network spatial structure of JK and LM was destroyed, so it could not absorb more oil. Therefore, OHC of LM and JK showed a trend of rising first and then falling.



**Figure 3.** Effect of BM on the apparent structure of three citrus fibers ((A) was FS, (B) was JK, and (C) was LM). The magnifications of a–d (0, 2, 6, and 10 h) and e–h (0, 2, 6, and 10 h) were 500 $\times$  and 2000 $\times$ , respectively.

### 3.5. Analysis of WSC

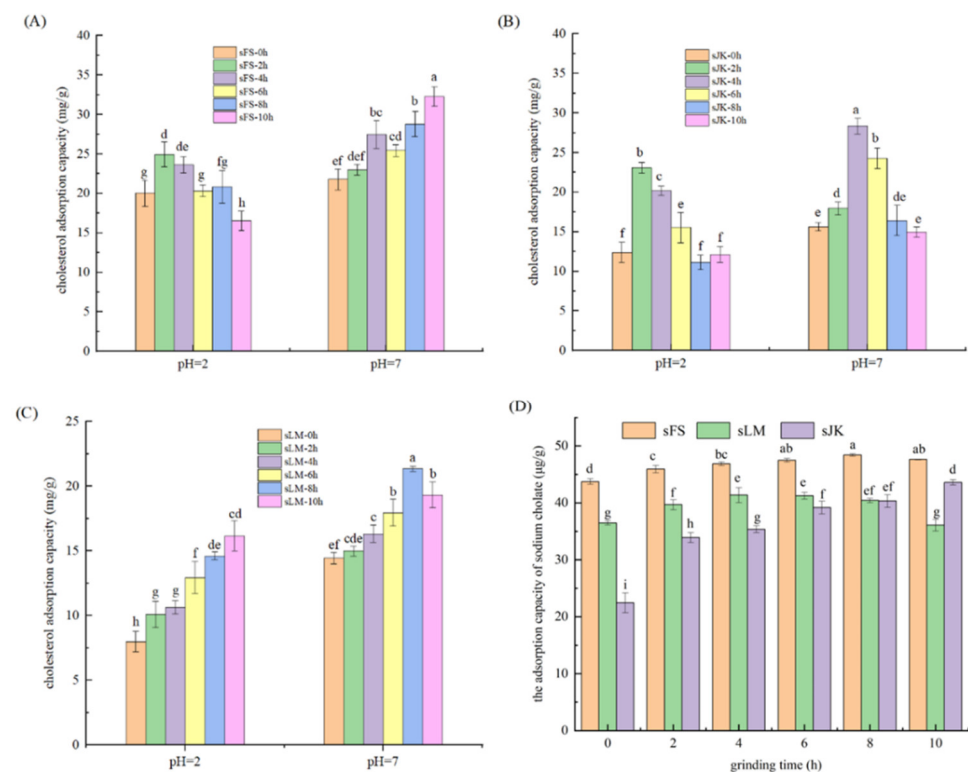
Citrus fiber has WSC, probably due to its loose network space structure with hydrophilic groups, which can absorb a certain amount of water [44]. The effects of BM on WSC of three citrus fibers are shown in Figure 2C. WSC and WHC of three citrus fibers showed the same change trend.



Compared with the unmodified samples, WSC of citrus fibers LM and JK increased by 9.28% and 12.45%, respectively, after 2 h of BM ( $p < 0.05$ ). After milling for 10 h, WSC of citrus fiber FS was increased by 38.3% ( $p < 0.05$ ). Furthermore, the porosity and specific surface area of LM and JK were increased after BM for 2 h, and more hydrophilic groups were exposed [16], so its WSC could be improved. However, after BM for 4–10 h, the side chain structure and spatial structure of the cellulose were destroyed, leading to the decrease in WSC of LM and JK. In addition, WSC of FS that contained high water-soluble dietary fiber increased with the increase in BM time, due to the fact that its hydrophilic groups in water-soluble dietary fiber were exposed. More polar groups and water binding sites of citrus fiber were exposed after BM, resulting in an increase in WSC of FS [45]. The rice bran was crushed by a grater and then the coarse powder was obtained through an 80-mesh sieve. The fine powders and superfine powders were further obtained by regulating the grinding time. The result proved that WSC of superfine powders from rice bran dietary fiber was obviously higher than that of its coarse powders and fine powders [18]. Chitrakar et al. also found that WSC of dietary fiber powders from asparagus leaves increased from 25.74% to 32.47% with the increase in grinding time [41]. This is because the porous matrix structure of cell wall polysaccharides was damaged by BM, which enables them to retain water through hydrogen bonds [46]. This is similar to the mechanism of increased WHC of citrus fibers.

### 3.6. Analysis of CAC

The influence of BM on CAC of three citrus fibers FS, JK, and LM is shown in Figure 4A–C. CAC can be associated with porosity and available surface area of dietary fibers [47].



**Figure 4.** Effect of BM on cholesterol adsorption capacity (A–C) and the adsorption capacity of sodium fibrous cholate (D) of three citrus fibers (JK, LM, and FS). Error bars represent the standard deviation of the mean of triplicate experiments. Different letters (a–i) indicate significant difference ( $p < 0.05$ ).

After BM, CAC of three citrus fibers was significantly increased at pH 7.0 and pH 2.0 ( $p < 0.05$ ). Among them, CAC of citrus fiber FS was the strongest as FS had more SDF. The internal structure of SDF had many pores and exhibited a strong adsorption capacity for cholesterol. Pectin and other SDF can improve the viscosity of foods in the intestinal tract, which can further absorb a large amount of cholesterol [48]. Compared with the unmodified samples, CAC of FS and LM at pH 7.0 increased by 48.34% and 47.95%, respectively, after 10 h of BM. CAC of JK increased by 41.50% after 6 h of BM. Because of the loose structure of citrus fibers, there were many holes on the surface after BM, which probably provided more binding sites for cholesterol [3].

In addition, CAC of three citrus fibers treated with pH 2.0 was lower than those treated with pH 7.0. Under acidic conditions, citrus fiber and cholesterol had partial positive charges and had a repulsive force, leading to the weakening of the binding force between citrus fiber and cholesterol [3], thus reducing CAC.

### 3.7. Analysis of Adsorption Capacity of Sodium Cholate

The influence of BM on the sodium cholate adsorption capacity of three citrus fibers is shown in Figure 4D. The adsorption capacity of sodium cholate of citrus fiber FS was the strongest, followed by that of LM and JK. After BM, the adsorption capacity of sodium cholate of three citrus fibers increased significantly ( $p < 0.05$ ). Compared with the unmodified samples, the sodium cholate adsorption capacity of FS and JK increased by 10.74% and 48.64% after BM for 10 h; and that of LM increased by 13.07% ( $p < 0.05$ ) after milling for 8 h. This was potentially due to the increase in SDF contents of three citrus fibers by BM, and the release of some chemical groups that adsorbed sodium cholate, which improved adsorption capacity of sodium cholate. Dietary fiber can effectively absorb sodium cholate, thus reducing the concentration of bile acid in the intestinal tract. As a result, the absorption of cholesterol was accelerated, and the blood glucose was then reduced in the body [49].

### 3.8. The Apparent Structure

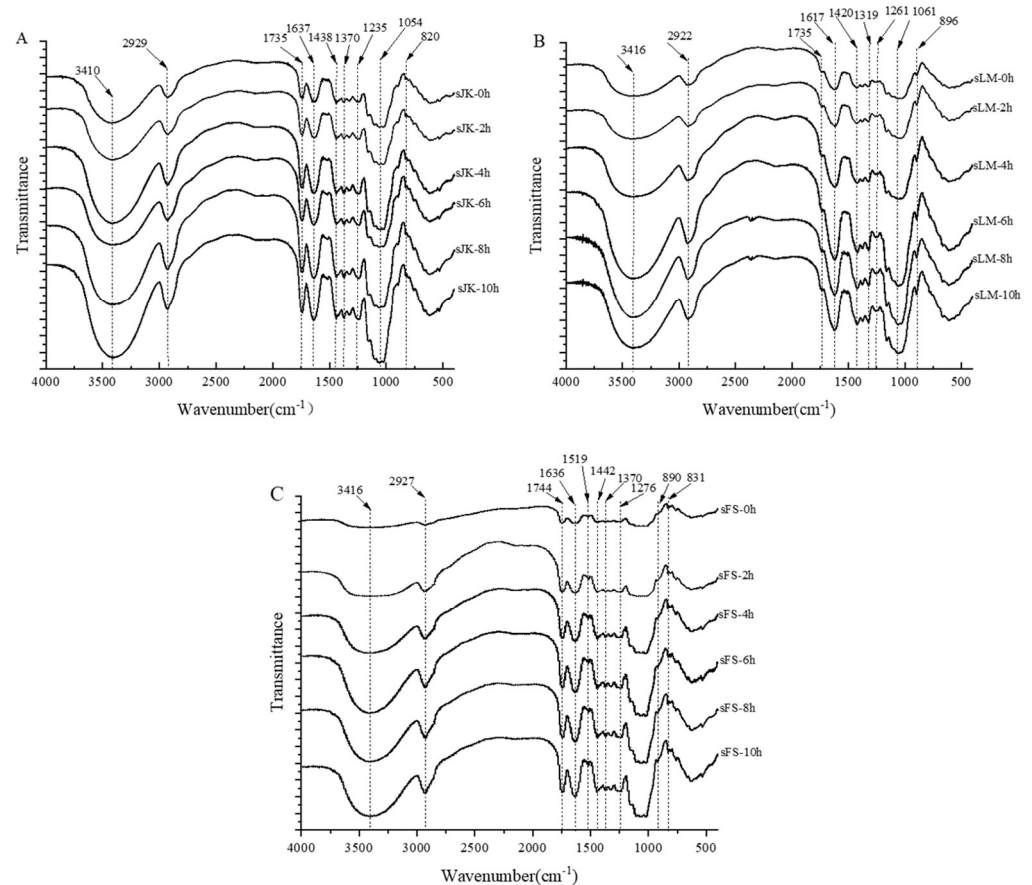
The effects of BM (0–10 h) on the apparent structure of FS, JK, and LM are shown in Figure 3A–C, respectively. It can be observed at  $500\times$  magnification that, before, BM, LM, and JK had a spherical structure, whereas FS had a flake structure. After BM for 2 h, JK and LM changed from a spherical to a fragmented conformation. The flake structures of three citrus fibers became smaller and more uniform in the grinding time between 2 and 10 h. The authors of [41] also studied the effect of low-temperature BM on microstructural characteristics of asparagus leaf by-product. SEM demonstrated that the particles were irregular and broken from large particles to small particles in the process of BM.

It was observed at  $2000\times$  magnification that, before BM, compared with FS and JK, the overall structure of LM was looser and had more pores. In addition, the surface of JK and LM was looser, and the number of pores increased after BM for 2 h, which exposed more hydrophilic and lipophilic groups in citrus fiber [41], leading to improve its functional properties such as WHC and OHC. The surface of JK and LM was smooth at BM 6–10 h, while the surface of FS showed more pores, so the functional properties of FS increased with the extension in the grinding time.

### 3.9. Analysis of FTIR

FTIR is an important indicator for evaluating proteins, fibers, and other substances [50,51]. Figure 5A–C respectively represent the effects of BM on FTIR of three citrus fibers. FTIR results of three citrus fibers were similar, but the curve transmittance results of the three citrus fiber samples were different. The peak vibration intensity of the three citrus fibers increased after BM due to the increase in the exposed -OH content in citrus fibers by BM [44]. As BM increased from 0 to 10 h, the peak values of three citrus fibers were enhanced in  $2927\text{ cm}^{-1}$  bands (stretching vibration of C-H on the polysaccharide methylene),  $1735\text{ cm}^{-1}$  and  $1636\text{ cm}^{-1}$  bands (vibration of acetyl ester and uronic acid groups on the hemicellulose), and  $1370\text{ cm}^{-1}$  bands (bending vibration of C-H in cellulose). The results showed that BM

can loosen the structure of citrus fibers, and more functional groups were exposed. In addition, the characteristic bending or tensile vibration peak of lignin at 1519 and 1276  $\text{cm}^{-1}$  did not decrease after BM, meaning that lignin was not degraded by BM [52].



**Figure 5.** Effect of BM on infrared spectra of three citrus fibers ((A) was JK, (B) was LM and (C) was FS).

#### 4. Conclusions

This study was the first to compare the effects of BM on structure, physicochemical, and functional properties of three citrus fibers (LM, JK, and FS) from different sources. The results showed that the particle size of three citrus fibers reduced significantly after BM, and more hydrophilic and lipophilic groups were exposed, improving WHC, OHC, and WSC of citrus fibers. In addition, the content of SDF of LM, JK, and FS increased, and the adsorption capacity of cholesterol and sodium cholate was improved. Finally, it can be concluded that FS treated by BM for 8 h has good functional and adsorption properties. BM is a green mechanical grinding technology with the advantages of environmental protection and low cost. It is an effective technology to improve the characteristics of citrus fibers, and can thus widen the application of citrus fibers in the food industry as natural functional ingredients.

**Author Contributions:** Conceptualization, Z.J. and M.Z.; methodology, Z.J.; software, Z.J.; validation, M.Z., Y.H. and C.M.; formal analysis, S.M.; investigation, Z.J.; resources, Z.J.; data curation, H.L. and X.L.; writing—original draft preparation, Z.J.; writing—review and editing, Z.J.; visualization, Y.M. and Y.L.; supervision, Z.J.; project administration, J.H.; funding acquisition, Z.J. All authors have read and agreed to the published version of the manuscript.

**Funding:** This study was supported by project for Natural Key Science Foundation of Heilongjiang Province of China (No. ZD2021C007) and Key Program of Heilongjiang Province of China (No. 2019ZX07B02-04).

**Data Availability Statement:** Data are contained within the article.

**Acknowledgments:** We thank the Key Laboratory of Dairy Science at Northeast Agricultural University for providing the facility support.

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

## References

1. Liu, N.; Xia, L.; Ping, Z.; Xueqian, Z.; Ou, Q.; Luqi, H.; Lanping, G.; Wenyuan, G. A Review of Chemical Constituents and Health-Promoting Effects of Citrus Peels. *Food Chem.* **2021**, *65*, 130585. [[CrossRef](#)] [[PubMed](#)]
2. Mahato, N.; Sharma, K.; Sinha, M.; Baral, E.R.; Koteswararao, R.; Dhyani, A.; Cho, M.H.; Cho, S. Bio-Sorbents, Industrially Important Chemicals and Novel Materials from Citrus Processing Waste as a Sustainable and Renewable Bioresource: A Review. *J. Adv. Res.* **2020**, *23*, 61–82. [[CrossRef](#)] [[PubMed](#)]
3. Song, L.W.; Qi, J.R.; Liao, J.S.; Yang, X.Q. Enzymatic and Enzyme-Physical Modification of Citrus Fiber by Xylanase and Planetary Ball Milling Treatment. *Food Hydrocoll.* **2021**, *121*, 107015. [[CrossRef](#)]
4. Hua, M.; Lu, J.; Qu, D.; Liu, C.; Zhang, L.; Li, S.; Chen, J.; Sun, Y. Structure, Physicochemical Properties and Adsorption Function of Insoluble Dietary Fiber from Ginseng Residue: A Potential Functional Ingredient. *Food Chem.* **2019**, *286*, 522–529. [[CrossRef](#)] [[PubMed](#)]
5. Liu, Z.; Zhou, X.; Wang, W.; Gu, L.; Hu, C.; Sun, H.; Xu, C.; Hou, J.; Jiang, Z. *Lactobacillus Paracasei* 24 Attenuates Lipid Accumulation in High-Fat Diet-Induced Obese Mice by Regulating the Gut Microbiota. *J. Agric. Food Chem.* **2022**, *15*, 4631–4643. [[CrossRef](#)] [[PubMed](#)]
6. Liu, Z.; Zhao, J.; Sun, R.; Wang, M.; Wang, K.; Li, Y.; Shang, H.; Hou, J.; Jiang, Z. *Lactobacillus Plantarum* 23-1 Improves Intestinal Inflammation and Barrier Function through the Tlr4/Nf-Kb Signaling Pathway in Obese Mice. *Food Funct.* **2022**, *13*, 5971–5986. [[CrossRef](#)]
7. Figuerola, F.; Hurtado, M.L.; Estévez, A.M.; Chiffelle, I.; Asenjo, F. Fibre Concentrates from Apple Pomace and Citrus Peel as Potential Fibre Sources for Food Enrichment. *Food Chem.* **2005**, *3*, 395–401. [[CrossRef](#)]
8. Mudgil, D.; Barak, S. Composition, Properties and Health Benefits of Indigestible Carbohydrate Polymers as Dietary Fiber: A Review. *Int. J. Biol. Macromol.* **2013**, *61*, 1–6. [[CrossRef](#)] [[PubMed](#)]
9. He, Y.; Wang, B.; Wen, L.; Wang, F.; Yu, H.; Chen, D.; Su, X.; Zhang, C. Effects of Dietary Fiber on Human Health. *Food Sci. Hum. Wellness* **2022**, *1*, 1–10. [[CrossRef](#)]
10. Dervisoglu, M.; Yazici, F. Note. The Effect of Citrus Fibre on the Physical, Chemical and Sensory Properties of Ice Cream. *Food Sci. Technol. Int.* **2006**, *2*, 159–164. [[CrossRef](#)]
11. Ozturk, O.K.; Mert, B. The Use of Microfluidization for the Production of Xanthan and Citrus Fiber-Based Gluten-Free Corn Breads. *LWT* **2018**, *96*, 34–41. [[CrossRef](#)]
12. Gedikoğlu, A.; Clarke, A.D. Quality Attributes of Citrus Fiber Added Ground Beef and Consumer Acceptance of Citrus Fiber Added Turkish Meat-Balls. *Food Health* **2019**, *5*, 205–214. [[CrossRef](#)]
13. Fernández-Ginés, J.M.; Fernández-López, J.; Sayas-Barberá, E.; Sendra, E.; Pérez-Alvarez, J.A. Effect of Storage Conditions on Quality Characteristics of Bologna Sausages Made with Citrus Fiber. *J. Food Sci.* **2003**, *2*, 710–714. [[CrossRef](#)]
14. Wang, L.; Shen, C.; Li, C.; Chen, J. Physicochemical, Functional, and Antioxidant Properties of Dietary Fiber from Rosa Roxburghii Tratt Fruit Modified by Physical, Chemical, and Biological Enzyme Treatments. *J. Food Process. Preserv.* **2020**, *44*, e14858. [[CrossRef](#)]
15. 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**, *4*, 586–593. [[CrossRef](#)]
16. Zhu, F.; Du, B.; Xu, B. Superfine Grinding Improves Functional Properties and Antioxidant Capacities of Bran Dietary Fibre From Qingke (Hull-Less Barley) Grown in Qinghai-Tibet Plateau, China. *J. Cereal Sci.* **2015**, *65*, 43–47. [[CrossRef](#)]
17. Chau, C.F.; Wen, Y.L.; Wang, Y.T. Effects of Micronisation on the Characteristics and Physicochemical Properties of Insoluble Fibres. *J. Sci. Food Agric.* **2006**, *14*, 2380–2386. [[CrossRef](#)]
18. Zhao, G.; Zhang, R.; Dong, L.; Huang, F.; Tang, X.; Wei, Z.; Zhang, M. Particle Size of Insoluble Dietary Fiber from Rice Bran Affects its Phenolic Profile, Bioaccessibility and Functional Properties. *LWT* **2018**, *87*, 450–456. [[CrossRef](#)]
19. Ye, F.; Tao, B.; Liu, J.; Zou, Y.; Zhao, G. Effect of Micronization on the Physicochemical Properties of Insoluble Dietary Fiber from Citrus (*Citrus Junos* Sieb. Ex Tanaka) Pomace. *Food Sci. Technol. Int.* **2016**, *3*, 246–255. [[CrossRef](#)]
20. Zhou, X.; Zhao, J.; Zhao, X.; Sun, R.; Sun, C.; Hou, D.; Zhang, X.; Jiang, L.; Hou, J.; Jiang, Z. Oil Bodies Extracted from High-Oil Soybeans (*Glycine Max*) Exhibited Higher Oxidative and Physical Stability than Oil Bodies from High-Protein Soybeans. *Food Funct.* **2022**, *6*, 3271–3282. [[CrossRef](#)]
21. Li, J.; Fu, J.; Ma, Y.; He, Y.; Fu, R.; Qayum, A.; Jiang, Z.; Wang, L. Low Temperature Extrusion Promotes Transglutaminase Cross-Linking of Whey Protein Isolate and Enhances its Emulsifying Properties and Water Holding Capacity. *Food Hydrocoll.* **2022**, *125*, 107410. [[CrossRef](#)]
22. Suryanti, V.; Kusumaningsih, T.; Rumingtyas, Y.S. Physicochemical Properties of Dietary Fibers from Artocarpus Camansi Fruit. *Pap. Presented IOP Conf. Ser. Mater. Sci. Eng.* **2017**, *193*, 012012. [[CrossRef](#)]

23. Speroni, C.S.; Bender, A.B.B.; Stiebe, J.; Ballus, C.A.; Ávila, P.F.; Goldbeck, R.; Morisso, F.D.P.; da Silva, L.P.; Emanuelli, T. Granulometric Fractionation and Micronization: A Process for Increasing Soluble Dietary Fiber Content and Improving Technological and Functional Properties of Olive Pomace. *LWT* **2020**, *130*, 109526. [[CrossRef](#)]
24. Wang, L.; Xu, H.; Yuan, F.; Pan, Q.; Fan, R.; Gao, Y. Physicochemical Characterization of Five Types of Citrus Dietary Fibers. *Biocatal. Agric. Biotechnol.* **2015**, *2*, 250–258. [[CrossRef](#)]
25. Dong, J.L.; Wang, L.; Lü, J.; Zhu, Y.Y.; Shen, R.L. Structural, Antioxidant and Adsorption Properties of Dietary Fiber from Foxtail Millet (*Setaria Italica*) Bran. *J. Sci. Food Agric.* **2019**, *8*, 3886–3894. [[CrossRef](#)]
26. Li, J.; Liu, Y.; Li, T.; Gantumur, M.A.; Qayum, A.; Bilawal, A.; Jiang, Z.; Wang, L. Non-Covalent Interaction and Digestive Characteristics Between A-Lactalbumin and Safflower Yellow: Impacts of Microwave Heating Temperature. *LWT* **2022**, *159*, 113206. [[CrossRef](#)]
27. Xu, C.; Fu, Y.; Liu, F.; Liu, Z.; Ma, J.; Jiang, R.; Song, C.; Jiang, Z.; Hou, J. Purification and Antimicrobial Mechanism of a Novel Bacteriocin Produced by *Lactobacillus Rhamnosus* 1.0320. *LWT* **2021**, *137*, 110338. [[CrossRef](#)]
28. Deng, M.; Lin, Y.; Dong, L.; Jia, X.; Shen, Y.; Liu, L.; Chi, J.; Huang, F.; Zhang, M.; Zhang, R. Physicochemical and Functional Properties of Dietary Fiber from Pummelo (*Citrus Grandis*, L. Osbeck) and Grapefruit (*Citrus Paradisi* Mcfad) Cultivars. *Food Biosci.* **2021**, *40*, 100890. [[CrossRef](#)]
29. Marín, F.R.; Soler-Rivas, C.; Benavente-García, O.; Castillo, J.; Pérez-Alvarez, J.A. By-Products from Different Citrus Processes as a Source of Customized Functional Fibres. *Food Chem.* **2007**, *2*, 736–741. [[CrossRef](#)]
30. 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* **2020**, *117*, 108652. [[CrossRef](#)]
31. Gao, W.; Chen, F.; Wang, X.; Meng, Q. Recent Advances in Processing Food Powders by Using Superfine Grinding Techniques: A Review. *Compr. Rev. Food Sci. Food Saf.* **2020**, *4*, 2222–2255. [[CrossRef](#)] [[PubMed](#)]
32. Wang, W.; Wang, M.; Xu, C.; Liu, Z.; Gu, L.; Ma, J.; Jiang, L.; Jiang, Z.; Hou, J. Effects of Soybean Oil Body as a Milk Fat Substitute on Ice Cream: Physicochemical, Sensory and Digestive Properties. *Foods* **2022**, *11*, 1504. [[CrossRef](#)] [[PubMed](#)]
33. Li, M.; Liu, Y.; Zhao, J.; Yu, R.; Hussain, M.A.; Qayum, A.; Jiang, Z.; Qu, B. Glycosylated Whey Protein Isolate Enhances Digestion Behaviors and Stabilities of Conjugated Linoleic Acid Oil in Water Emulsions. *Food Chem.* **2022**, *383*, 132402. [[CrossRef](#)] [[PubMed](#)]
34. Zhao, X.; Wang, K.; Zhao, J.; Sun, R.; Shang, H.; Sun, C.; Liu, L.; Hou, J.; Jiang, Z. Physical and Oxidative Stability of Astaxanthin Microcapsules Prepared with Liposomes. *J. Sci. Food Agric.* **2022**, *11*, 4909–4917. [[CrossRef](#)]
35. 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](#)]
36. Jiang, Z.; Mu, S.; Ma, C.; Liu, Y.; Ma, Y.; Zhang, M.; Li, H.; Liu, X.; Hou, J.; Tian, B. Consequences of Ball Milling Combined with High-Pressure Homogenization on Structure, Physicochemical and Rheological Properties of Citrus Fiber. *Food Hydrocoll.* **2022**, *127*, 107515. [[CrossRef](#)]
37. Ramachandraiah, K.; Chin, K.B. Evaluation of Ball-Milling Time on the Physicochemical and Antioxidant Properties of Persimmon by-Products Powder. *Innov. Food Sci. Emerg. Technol.* **2016**, *37*, 115–124. [[CrossRef](#)]
38. Jiang, G.; Ramachandraiah, K.; Wu, Z.; Li, S.; Eun, J.B. Impact of Ball-Milling Time on the Physical Properties, Bioactive Compounds, and Structural Characteristics of Onion Peel Powder. *Food Biosci.* **2020**, *36*, 100630. [[CrossRef](#)]
39. Ting, Y.; Jiang, Y.; Ho, C.T.; Huang, Q. Common Delivery Systems for Enhancing in Vivo Bioavailability and Biological Efficacy of Nutraceuticals. *J. Funct. Foods* **2014**, *7*, 112–128. [[CrossRef](#)]
40. Zhu, K.; Huang, S.; Peng, W.; Qian, H.; Zhou, H. Effect of Ultrafine Grinding on Hydration and Antioxidant Properties of Wheat Bran Dietary Fiber. *Food Res. Int.* **2010**, *4*, 943–948. [[CrossRef](#)]
41. Chitrakar, B.; Zhang, M.; Zhang, X.; Devahastin, S. Bioactive Dietary Fiber Powder from Asparagus Leaf by-Product: Effect of Low-Temperature Ball Milling on Physico-Chemical, Functional and Microstructural Characteristics. *Powder Technol.* **2020**, *366*, 275–282. [[CrossRef](#)]
42. Elleuch, M.; Bedigian, D.; Roiseux, O.; Besbes, S.; Blecker, C.; Attia, H. Dietary Fibre and Fibre-Rich by-Products of Food Processing: Characterisation, Technological Functionality and Commercial Applications: A Review. *Food Chem.* **2011**, *2*, 411–421. [[CrossRef](#)]
43. Huang, J.Y.; Liao, J.S.; Qi, J.R.; Jiang, W.X.; Yang, X.Q. Structural and Physicochemical Properties of Pectin-Rich Dietary Fiber Prepared from Citrus Peel. *Food Hydrocoll.* **2021**, *110*, 106140. [[CrossRef](#)]
44. Zhang, Y.; Liao, J.; Qi, J. Functional and Structural Properties of Dietary Fiber from Citrus Peel Affected by the Alkali Combined with High-Speed Homogenization Treatment. *LWT* **2020**, *128*, 109397. [[CrossRef](#)]
45. Chau, C.F.; Cheung, P.C. Effects of the Physico-Chemical Properties of Three Legume Fibers on Cholesterol Absorption in Hamsters. *Nutr. Res.* **1999**, *2*, 257–265. [[CrossRef](#)]
46. Kethireddipalli, P.; Hung, Y.C.; Phillips, R.D.; Mcwatters, K.H. Evaluating the Role of Cell Wall Material and Soluble Protein in the Functionality of Cowpea (*Vigna Unguiculata*) Pastes. *J. Food Sci.* **2002**, *1*, 53–59. [[CrossRef](#)]
47. Liu, Y.; Zhang, H.; Yi, C.; Quan, K.; Lin, B. Chemical Composition, Structure, Physicochemical and Functional Properties of Rice Bran Dietary Fiber Modified by Cellulase Treatment. *Food Chem.* **2021**, *342*, 128352. [[CrossRef](#)] [[PubMed](#)]
48. Liu, J.; Wang, Z.; Wang, Z.; Hao, Y.; Wang, Y.U.; Yang, Z.; Li, W.; Wang, J. Physicochemical and Functional Properties of Soluble Dietary Fiber from Different Colored Quinoa Varieties (*Chenopodium Quinoa* Willd). *J. Cereal Sci.* **2020**, *95*, 103045. [[CrossRef](#)]

49. Dziejczak, K.; Szwengel, A.; Górecka, D.; Gujska, E.; Kaczkowska, J.; Drożdżyńska, A.; Walkowiak, J. Effect of Wheat Dietary Fiber Particle Size During Digestion in Vitro on Bile Acid, Faecal Bacteria and Short-Chain Fatty Acid Content. *Plant Foods Hum. Nutr.* **2016**, *2*, 151–157. [[CrossRef](#)]
50. Ma, Y.; Liu, Y.; Yu, H.; Mu, S.; Li, H.; Liu, X.; Zhang, M.; Jiang, Z.; Hou, J. Biological Activities and in Vitro Digestion Characteristics of Glycosylated A-Lactalbumin Prepared by Microwave Heating: Impacts of Ultrasonication. *LWT* **2022**, *158*, 113141. [[CrossRef](#)]
51. Zhang, W.; Zhao, P.; Li, J.; Wang, X.; Hou, J.; Jiang, Z. Effects of Ultrasound Synergized with Microwave on Structure and Functional Properties of Transglutaminase-Crosslinked Whey Protein Isolate. *Ultrason. Sonochem.* **2022**, *83*, 105935. [[CrossRef](#)]
52. Tewari, J.C.; Dixit, V.; Cho, B.K.; Malik, K.A. Determination of Origin and Sugars of Citrus Fruits Using Genetic Algorithm, Correspondence Analysis and Partial Least Square Combined with Fiber Optic Nir Spectroscopy. *Spectrochim. Acta Part A Mol. Biomol. Spectrosc.* **2008**, *3*, 1119–1127. [[CrossRef](#)] [[PubMed](#)]