Heliyon 9 (2023) e21609

Contents lists available at ScienceDirect

Heliyon

journal homepage: www.cell.com/heliyon

Effects of pre-drying treatment and particle sizes on physicochemical and structural properties of pumpkin flour

Derese Wodajo Bekele [a,b,*], Shimelis Admassu Emire [a]

[a] School of Chemical and Bioengineering, Addis Ababa Institute of Technology, Addis Ababa, Ethiopia
[b] Department of Food Process Engineering, Wolkite University, Wolkite, Ethiopia

ARTICLE INFO

Keywords:
Compositional analysis
Particle size
Structural properties

ABSTRACT

The objective of this study is to investigate the effect of pre-drying treatment and particle size on pumpkin flour's compositional, functional, pasting, thermal, and structural properties. Pre-drying treatment and reducing the size of particles have led to an increase in the levels of crude fiber, phytochemicals, and functional properties. However, the amounts of moisture and lightness of color decreased with pretreatment, but improved with particle size reduction. Among the samples, those that were finely milled after pre-treatment showed the highest viscosities, while the untreated samples that were coarsely milled had lower viscosities. The pre-drying treatments yielded notable reductions in both gelatinization temperature and enthalpy of the flour matrix, in comparison to untreated flours. FTIR analysis has indicated that no new functional groups were produced. XRD analysis suggests that while pre-drying treatment leads to decreases in crystallinity index, fine milling leads to increments. The morphological pattern suggested that pre-treatment effectively altered the original surface structure of the flour, but particle size reduction did not.

1. Introduction

Pumpkins come in a variety of forms, dimensions, and colors. Because of its nutritional and health-protective polysaccharides, agriculture, food processing, pharmaceutical as well as feed industry have all taken an increasing interest in pumpkin fruit and pumpkin-derived products in recent years [1]. Pumpkin is a reliable source of beta-carotene, fibre, pectin, mineral salts, vitamins, and other substances that are beneficial to health [2]. Because of these facts, pumpkin is processed into a variety of foods. According to Que et al. [3], pumpkin flour is the major product of pumpkin fruit in processing since it can be stored for a long time and used in manufacturing formulated foods. However, drying conditions, including high temperature, light, and oxygen exposure, would cause degradation of carotenoids and thereby affect the final products' attractive color and nutritive value [4].

Pre-drying, on the other hand, can shorten drying durations while improving quality by retaining color and flavor and reducing nutrient loss and degradation through enzyme action [5]. It is suggested that the drying time be shortened in order to maintain the bioactive components of pumpkin during the flour conversion process. A previous study [6] discovered that ultrasonic, microwave, and combined pretreatment methods had better compositional, phytochemical, and functional qualities than untreated pumpkin flour. Ultrasound and microwave combined pre-drying treatment has been discovered to decrease drying time significantly. It results in a quicker and more effective drying process, while also improving the quality of the dried fruits by maintaining their nutritional

* Corresponding author. Wolkite University, Wolkite, Ethiopia.
E-mail address: derese.wodajo@wku.edu.et (D.W. Bekele).

https://doi.org/10.1016/j.heliyon.2023.e21609
Received 26 April 2023; Received in revised form 22 October 2023; Accepted 24 October 2023
Available online 1 November 2023
2405-8440/

properties [6]. Compared to other pretreatment methods, the use of ultrasound and microwave has been found to better retain vitamin C and total phenolic content [7]. Additionally, decreasing the particle size of the flour is an effective option to increase the bioactive availability, as larger particle sizes may be difficult to extract and may require longer heat treatment [8]. In addition, as reported by Lee and Yoon [9] and Zhu et al. [10], different variations in particle size would likewise affect changes in flour color and bioactive chemicals. Reduced particle size has been shown in several studies to increase the antioxidant activities of some food powders, including winter wheat (*Triticum aestivum* L.), red rice (Oryza sativa L.), and Qingke (hull-less barley) [11–13]. This could be a good way to increase the bioactivity and bioavailability of flour. So, to enhance its use in functional food products, it would be important to look for acceptable particle sizes for pumpkin flour. However, there has been a lack of previous research focused on investigating the combined impact of pre-drying treatment and particle size on the quality of pumpkin flour, which directly influences the overall quality of the flour. Therefore the goal of this work was to study the interaction effect of pre-drying treatment and particle size on physical, nutritional, phytochemical, functional, thermal, pasting, and structural properties of flours and to provide theoretical support for the application of pre-drying treatment and fine milling in the food industries.

2. Material and methods

2.1. Materials and chemicals

The fresh, healthy, ripped pumpkin was brought from a local supermarket (Wolkite town, Ethiopia). The Standards and reagents used were quercetin (≥98 %, sigma Aldric, Germany), 2,2-Diphenyl-1-picrylhydrazyl (Sigma Aldrich, Germany), gallic acid (97.5–100 % Sigma Aldrich, China), phytic acid sodium salt hydrate (Sigma Aldrich, Switzerland), (+-) Catechin hydrate (≥96.0 %, Sigma Aldrich, China), Folin-Ciocalteu's (2 N, Sigma Aldrich, USA), Vanillin (≥99.5 %, UNI-CHEM, Roth, France), Almunium Chloride (99 % Loba chemic, India), Methanol (M.wt. = 34.02 g/mol, Biochem chemopharma, France) and Sodium carbonate (≥99.5 % Carl Roth GmbH, Karlsruhe) and Nitric acid (69 % Loba chemic, India). All other chemical reagents were purchased locally and were of analytical grade.

2.2. Sample preparation

Untreated and pretreated (for 20 min in ultrasonic followed by 6 min at 300 W in the microwave) pumpkin slices (15 × 15 mm^3) were dried by a fluidized bed dryer at 60 °C for 180 and 121 min, respectively [14]. The sliced of dried pumpkin were milled coarsely by hammer mill (Model BH24 1DY, Armfield, England), and then the flours were screened through 500 μm sieves to separate granulates. The resulting coarser flours were micronized using ball-milling (Planetary type ball mill, PM 100; Restch, Germany) at 300r min^{-1} for 15 min three times with an interval of 30 min to avoid flour overheating. The milled flour was also split into distinct particles size fractions (250–150, 150–100, 100–75, and <75 μm particle size) using a set of screen sieved with the vibratory sieve shaker for 5 min. The milled flour obtained was stored at 4 °C in brown zipped bags until further analysis.

2.3. Physical property analysis

The sample color was detected via Hunter Lab Colorimeter, Minolta, using the hunter scale of L*, a*, and b × values as indicators. The water activity of pumpkin flour was measured by a water activity meter (HD-3A, NanBei, China) [15]. The pH and Total Soluble Solid were determined according to official methods of AOAC [16] and AOAC [17] using a pH meter (BANTE Multiparameter/China) and a digital refractometer (Model-A670, Hanon, China) at 25 °C, respectively.

2.4. Chemical properties analysis

Proximate analysis: The Moisture, protein, fat, and ash were determined in triplicates by Association of Official Analytical Chemists (AOAC) official methods 967.19, 920.165, 920–39, and 941.12 [18], respectively. The crude fiber was estimated by 920.169 AOAC [19] method, while Carbohydrate (CHO) was calculated by difference.

Phytochemical property analysis: First, the methanol extracts were prepared from pumpkin flour according to Ferreira et al. [20]. Ten grams of pumpkin flour were extracted with 100 mL of methanol at 25 °C at 150 rpm for 24 h using a temperature shaker incubator (ZHWY-103 B) and then clarified through Whatman No. 1 paper. The deposit was extracted with two additional 100 mL portions of methanol described above. The methanolic extracts were dried using a Rota evaporator (R-300, Buchi, Switzerland) at a temperature of 40 °C until there was no more solvent left. They were then dissolved again in methanol at a concentration of 50 mg/mL and kept at a temperature of 4 °C for future use. The total phenolic content was then assessed using the Folin-Ciocalteu method in triplicate at a wavelength of 735 nm with gallic acid as a standard, and the total flavonoid concentration was assessed using the colorimetric method at a wavelength of 510 nm with quercetin as a standard, in accordance with inuye et al. [21]. Antioxidant activities were assessed using the DPPH methods [22] by using ascorbic acid as a standard without extract or control. Total carotenoid was estimated based on the methodology of de Carvalho et al. [23]. Total carotenoids were expressed as μg per g of dry matter. All analysis was carried out in triplicate.

2.5. Functional properties analysis

Bulk density (g/mL) was established in accordance with Goula, Adamopoulos and Kazakis [24], water holding capacity (WHC) and WSI were estimated according to Zhao et al. [25], while swelling capacity (SW) and oil absorption capacity (OAC) were assessed based on the procedure outlined by Ye et al. [26]. The measurement of each sample was repeated three times.

2.6. Pasting properties

The pasting properties of the flours were evaluated using a rapid visco-analyzer (Perten RVA 4800, Perkins Elmer, Sweden). Pasting parameters were calculated by adjusting moisture to 15 % (using the flour's moisture content as a guide, extra distilled water was added to reach 15 % moisture) at 250 rpm speed with a starting temperature of 50 °C for 1 min, raising to 95 ± 0.1 °C in 3 min 42 s, and holding for 2 min 30 s. After the holding time, the temperature was ramped down to 50 ± 0.1 °C in 3 min 48 s by the cooling system [27].

2.7. Thermal properties

Thermal properties were analyzed by differential scanning calorimeter (SKZ1052B, Hunan, china). Using an empty aluminum pan as a reference, 8 mg of each sample were placed in a hermetic aluminum pan and heated from 20 to 150 °C at a rate of 10 °C/min in a 50 mL/min nitrogen flow. Onset temperature (To), peak temperature (Tp), conclusion temperature (Tc), and change in enthalpy (ΔH) were assessed [27] from the curve obtained.

2.8. Structural properties

Scanning electron microscope (SEM): The morphological characterization of flour particles was studied using a scanning electron microscope (JCM-6000 plus, Jeol Ltd., Korea) to study the effect of grinding and pretreatment on the structure modification. The sample was coated with a thin gold layer to make the samples conductive, and images were captured at an accelerated voltage of 5 kV at 2000 × magnification [28].

Fourier transform infrared (FT-IR) spectroscopy: FTIR spectra were collected using FTIR spectrometer (Nicolet is50 ABX, Thermofisher Scientific, German) over the range 4000–400 cm^{-1} by the potassium bromide (KBr) pellet method a spectral resolution of 4 cm^{-1} [29].

XRD Analysis: The crystallinity and structural properties of pumpkin flour of different sizes were characterized by an X-ray diffractometer (XRD-7000, Shangai Drawel scientific instrument co., Ltd., China) operating at 40 kV and 40 mA. The sample was dispersed onto a stub and placed within the chamber of an analytical X-ray diffractometer (wavelength = 1.54 Å, CuKα radiation). The diffraction data were collected in the 2θ range from 5 to 80° at a 5°/min scan rate [30].

2.9. Statistical analysis

Conventional statistical methods were used to calculate means and standard deviations. The software package SAS version 9.0 (SAS Institute, Inc., Cary, North Carolina, USA) using analysis of variance (ANOVA) was applied to the data to determine differences ($P < 0.05$). Tukey's HSD test at the significance level of 5 % ($P < 0.05$) was used to determine significant differences among flours. After characterizing their physical, chemical, and functional qualities, notably by looking into their fiber and phytochemical makeup, other

Table 1
Effects of particle size and pretreatment method interaction on Water activity, pH, TSS, and color properties of flour.

Sample code	Water activity	pH	TSS	L*	a*	b*
CON1	0.35 ± 0.04^{ab}	6.59 ± 0.32^{a}	4.28 ± 0.13^{a}	83.35 ± 0.56^{a}	4.55 ± 0.40^{d}	37.27 ± 1.42^{d}
CON	0.38 ± 0.03^{ab}	6.55 ± 0.30^{a}	4.21 ± 0.01^{a}	82.06 ± 1.15^{ab}	5.02 ± 0.60^{cd}	38.68 ± 2.00^{cd}
CON2	0.39 ± 0.02^{ab}	6.53 ± 0.24^{a}	4.13 ± 0.23^{a}	81.00 ± 092^{b}	5.67 ± 0.69^{bc}	39.17 ± 1.06^{cd}
CON3	0.41 ± 0.04^{a}	6.41 ± 0.16^{a}	4.03 ± 0.25^{a}	79.92 ± 0.93^{b}	5.77 ± 0.78^{bc}	39.58 ± 1.82^{c}
20UM1	0.31 ± 0.00^{b}	6.42 ± 0.15^{a}	4.07 ± 0.31^{a}	77.13 ± 1.69^{c}	6.03 ± 0.37^{b}	42.14 ± 1.03^{b}
20UM	0.37 ± 0.05^{ab}	6.35 ± 0.22^{a}	3.87 ± 0.62^{a}	76.03 ± 2.31^{cd}	7.19 ± 0.22^{a}	43.64 ± 0.74^{ab}
20UM2	0.38 ± 0.04^{ab}	6.31 ± 0.04^{a}	3.76 ± 0.24^{a}	74.78 ± 1.51^{d}	7.56 ± 0.13^{a}	44.13 ± 1.05^{ab}
20UM3	0.41 ± 0.03^{ab}	6.26 ± 0.04^{a}	3.67 ± 0.44^{a}	73.94 ± 0.95^{d}	7.61 ± 0.20^{a}	44.51 ± 0.91^{a}
Effect						
Pretreatment (p)	NS	***	***	***	***	***
Particle size (pz)	***	NS	NS	***	***	***
P × pz	***	NS	NS	***	***	***

Values are mean ± standard deviation. Means that sharing the same letters in columns is not significantly different from each other (Tukey's HSD test, $p < 0.05$). *** Significant effect at $p < 0.05$, NS, Not significant at $p < 0.05$. CON1, control flour, particle size is < 75 μm; CON, control flour, particle size is 75–100 μm; CON2, control flour, particle size is 100–150 μm; CON3, control flour, particle size is 150–250 μm; 20UM1, pretreated flour, particle size is < 75 μm; 20UM, pretreated flour, particle size is 75–100 μm and; 20UM2, pretreated flour, particle size is 100–150 μm; 20UM3, pretreated flour, particle size is 150–250 μm; TSS, total soluble solid; L*, whiteness; a*, redness; b*, yellowness.

flour features, such as pasting, thermal, and structural characteristics, were analyzed with the aim of using the flour for functional foods.

3. Result and discussion

3.1. Physical properties

The physical properties of all flours are presented in Table 1. Even though water activity values increased with the increasing particle size of the flour, it was found to be not significant ($P < 0.05$) except for 20UM1 (0.31 + 0.00) and CON3 (0.41 + 0.04) flour samples. When it comes to extended storage of food products, manufacturers and formulators commonly use aw values to forecast moisture migration and assess how it might impact the product's properties [31].

The water activity of flour can decrease with reducing particle size because of increase in surface area-to-volume ratio of the flour particles. When the particle size of flour is reduced, the surface area of the flour particles increases, while the volume decreases. This higher surface area can lead to more water molecules being adsorbed onto the surface of the flour particles, which can result in a lower water activity [32].

The pH, which classifies the samples as acidic or slightly acidic flours, and total soluble solids (TSS) values were slightly increased with decreasing size of the flour but not significant at $P < 0.05$. While CON1 showed higher pH and TSS of 6.59 + 0.32 and 4.28 + 0.13 but 20UM3 flour showed lower in both pH (6.26 + 0.04) and TSS (3.67 + 0.44). The higher TSS in fine particle size could be because of the surface area to volume ratio increases. More contact with the solvent is made possible by the bigger surface area, increasing solubility.

Color is an important quality that affects consumer choice of foods [33]. The data indicate that the hunter color is significantly different due to pretreatment, particle size, and interaction between both. While color lightness (L) varied from 83.35 to 77.13, a × and b × values ranged from 7.61 to 4.55 and 44.51 to 37.27, respectively. The lightness increases with particle size reduction and decreases with pre-drying treatment, but b × values were in the opposite direction. According to the information presented in Table 1, the lightness of untreated flour is higher than that of pretreated flours milled to the same particle size, and also they showed significant differences between groups ($p < 0.05$). The lowest L* value was recorded for the 20UM3 (73.94 + 0.95) flour, while the highest was obtained for CON1 (83.35 + 0.56) flour. As particle size decreases, a × and b × values of pretreated and untreated flour decrease, with the highest value noted for 20UM3 flour and the lowest for the CON1 flour sample. The increased lightness of the sample may be due to a combination of factors, including a lower protein content in finer particle size [34] and higher surface area of the particles, which increases the reflection of light [35,36]. In another way, while pre-drying treatment preserves the loss of color pigment, the exposure of internal materials during fine milling could contribute to brightness improvement [37,38]. Thus implied that the degree of yellowness was lowered in untreated flour as compared to pretreated sample, which could be due to loss of pigmentation during the drying process, while further decreases of a × and b × values for both pretreated and untreated samples could be possibly due to effects of grinding and sieving. Ahmed, Al-attar and Arfat [35] and Ahmed et al. [27] reported a similar case in L*, a*, and b × values for water chestnut flour, lentil flour, and rice flour milled to different particle sizes. On contrary to this study, the lightness (L*) and yellowness (b*) of mango peel powder increased with particle size but greenness (a*) decreased [39].

3.2. Proximate

As shown in Table 2, moisture, fat, and crude fibre content increase with a reduction in particle size, but ash, protein, and carbohydrate content decrease. Nutritional content was enhanced due to pretreatment except for moisture and carbohydrate content. The

Table 2
Effects of interaction between particle size and pretreatment on proximate composition of pumpkin flour.

Sample code	Moisture	Ash	Crude Fat	Crude protein	Crude fibre	Carbohydrate
CON1	10.61 ± 0.55^{a}	5.22 ± 0.33^{a}	1.59 ± 0.26^{a}	6.99 ± 0.85^{d}	12.79 ± 1.08^{ab}	62.79 ± 1.29^{abc}
CON	10.18 ± 0.35^{ab}	5.34 ± 0.49^{a}	1.47 ± 0.21^{a}	7.30 ± 0.81^{cd}	12.36 ± 1.08^{ab}	63.35 ± 2.38^{abc}
CON2	9.22 ± 0.99^{abc}	5.45 ± 0.48^{a}	1.33 ± 0.70^{a}	7.88 ± 0.91^{bcd}	12.00 ± 0.94^{b}	64.12 ± 1.36^{ab}
CON3	8.82 ± 0.79^{cb}	5.68 ± 0.41^{a}	1.18 ± 0.32^{a}	8.20 ± 0.49^{bcd}	11.03 ± 0.29^{b}	65.10 ± 0.88^{a}
20UM1	9.82 ± 0.31^{ab}	5.44 ± 0.58^{a}	1.93 ± 0.19^{a}	9.05 ± 0.44^{abc}	14.39 ± 0.40^{a}	59.37 ± 1.06^{c}
20UM	9.25 ± 0.56^{abc}	5.57 ± 0.54^{a}	1.78 ± 0.24^{a}	9.56 ± 0.49^{ab}	13.12 ± 0.24^{ab}	60.72 ± 1.15^{bc}
20UM2	8.57 ± 0.49^{cb}	5.88 ± 0.43^{a}	1.74 ± 0.18^{a}	10.31 ± 0.46^{a}	12.80 ± 0.65^{ab}	60.70 ± 1.53^{bc}
20UM3	7.86 ± 0.44^{c}	6.00 ± 0.25^{a}	1.71 ± 0.19^{a}	10.66 ± 0.64^{a}	12.54 ± 0.73^{ab}	61.23 ± 1.16^{abc}
Effect						
Pretreatment (p)	***	NS	***	***	***	***
Particle size (pz)	***	NS	NS	***	***	***
P × pz	***	Ns	NS	***	***	***

Values are mean ± standard deviation. This means sharing the same letters in columns is not significantly different from each other (Tukey's HSD test, $p < 0.05$). *** Significant effect at $p < 0.05$, NS, Not significant at $p < 0.05$. CON1, control flour, particle size is < 75 μm; CON, control flour, particle size is 75–100 μm; CON2, control flour, particle size is 100–150 μm; CON3, control flour, particle size is 150–250 μm; 20UM1, pretreated flour, particle size is < 75 μm; 20UM, pretreated flour, particle size is75–100 μm and; 20UM2, pretreated flour, particle size is 100–150 μm; 20UM3, pretreated flour, particle size is 150–250 μm.

lowest moisture content was found in the bigger, pretreated particle size 20UM3 (7.86 + 0.44 %), whereas the highest value was found in the smaller, untreated particle size CON1 (10.61 + 0.55). The possible reason is due to ultrasound pre-drying treatment increases the water permeability due to the formation of microchannels as a result of expansion and contraction in the food matrix [40]. Also, Önal et al. [41] reported that microwave heating penetrates the foodstuff quickly and increases the product temperature and, consequently, rapid water evaporation.

In addition, smaller particle sizes absorb more moisture from the atmosphere due to higher surface area than larger particle sizes, thus increasing moisture content. The top three flour samples in carbohydrate and protein content were CON3 (65.10 + 0.88), CON2 (64.12 + 1.36 %), CON1 (63.35 + 2.38), and 20UM3 (10.66 + 0.64), 20UM2 (10.31 + 0.46), and 20UM (9.56 + 0.49), respectively. Also, as depicted in Table 2, there is no significant difference between flour samples ($p < 0.05$) in ash and fat content due to the interaction between pretreatment and particle size. Pretreated pumpkin slice with a particle size of <75 μm had a crude fiber content that was only significantly ($p < 0.05$) greater than other flours, whereas untreated pumpkin slice with a particle size of 150–250 μm had a lower value (11.03 + 0.29 %). This value was higher than that of Farombi and Oyekanmi [42] and Adelerin, Ifesan and Awolu [43] findings for pumpkin pulp and boiled pumpkin pulp flour, 11.46 + 0.10 % and 13.22 + 0.03, respectively. The increase in crude fiber content with pretreatment may be due to the breakdown of cell walls and the release of fiber during the drying process [44]. When the size of flour particles is reduced, the surface area of the particles increases and this can result in a greater exposure of the fiber-containing cell walls. As a result, the crude fiber content of the flour increases [45]. Protein content increases with pre-drying treatment and the possible reason could be due to increasing the penetration of solvent into cellular material [46]. Pre-drying also enhance the removal of moisture from the fruit before it is ground into flour, which can lead to a concentration of protein in the resulting flour. Also, the smaller the size of a particle, the lower the protein content of that flour. The reason for the decrease in protein content during particle size reduction might be that the mechanical forces and heat produced during this process can cause the protein to undergo denaturation and aggregation, resulting in a loss of protein content [45]. Similar findings were reported by Ahmed et al. [27] and Ahmed, Al-attar and Arfat [35] in lentil flour and water chestnut flour, respectively.

Carbohydrates increase with an increase in particle size but decrease with pre-drying treatment. Since carbohydrate was calculated by the difference method, their lower value might be due to the higher value of other parameters. The result is also in line with the value observed for water chestnut flour with an increase in particle size [35]. This study reveals that particle size did not influence the fat content, which is similar to that observed by Ahmed et al. [27] for Indian and Turkish lentil flour, but contrary to that observed in water chestnut flour [35]. As stated by Luthria, Noel and Vinjamoori [47], the decrease in crude fat extraction in larger particle-size flours could be easily attributed to the lower surface area, which decreases the extraction efficiency. Both pre-drying treatment and milling did not influence the ash content.

3.3. Phytochemicals

As shown in Table 3, flour total phenol, total flavonoid, and total carotenoid concentrations showed slightly significant differences ($p < 0.05$) due to the interaction between pre-drying treatment and particle size reduction.

However, there is a noticeable difference between untreated and pretreated flour at the same particle size, high in content with pre-drying treatment, and smaller particle size. Total phenol and carotenoid contents were not significantly different between the pre-treated pumpkin slice milled to different particle sizes.

According to Tekin and Baslar [48], the strong shear force generated by the cavitation effect of ultrasound treatment damaged the cell walls and promoted the release of active ingredients in the material. Regarding total phenol, flavonoids, and carotenoids, 20UM1 flour sample had the highest amount (6.52 mgGAE/g, 1.92 mgCE/g, and 139.79 mg/g), whereas CON3 flour had the lowest (3.43 mgGAE/g, 0.99 mgCE/g, and 57.90 mg/g), respectively. The result agreed with the investigation of Zhao et al. [49] for red grape

Table 3
Effects of interaction between particle size and pretreatment on phytochemical properties of pumpkin flour.

Sample code	Total Phenol (mgGAE/g)	Total Flavonoids (mgCE/g)	Total carotenoid (μg/g)
CON1	3.79 ± 0.15^{b}	1.28 ± 0.66^{c}	77.93 ± 8.35 b
CON	3.59 ± 0.59^{b}	1.15 ± 0.52^{cd}	69.85 ± 2.00BCE
CON2	3.47 ± 0.68^{b}	1.04 ± 0.23^{d}	63.43 ± 4.03BCE
CON3	3.43 ± 0.32^{b}	0.99 ± 0.79^{d}	57.90 ± 7.42c
20UM1	6.52 ± 0.59^{a}	1.92 ± 0.85^{a}	139.74 ± 0.96a
20UM	6.39 ± 0.67^{a}	1.85 ± 0.95^{ab}	133.37 ± 2.39a
20UM2	6.37 ± 0.49^{a}	1.74 ± 0.59^{ab}	132.84 ± 1.83a
20UM3	6.34 ± 0.88^{a}	1.72 ± 0.43^{b}	132.03 ± 8.38a
Effect			
Pretreatment (p)	***	***	***
Particle size (pz)	***	***	***
P × pz	***	***	***

Values are mean ± standard deviation. Means that sharing the same letters in columns is not significantly different from each other (Tukey's HSD test, $p < 0.05$). *** Significant effect at $p < 0.05$, NS, Not significant at $p < 0.05$. CON1, control flour, particle size is < 75 μm; CON, control flour, particle size is 75–100 μm; CON2, control flour, particle size is 100–150 μm; CON3, control flour, particle size is 150–250 μm; 20UM1, pretreated flour, particle size is < 75 μm; 20UM, pretreated flour, particle size is 75–100 μm and; 20UM2, pretreated flour, particle size is 100–150 μm; 20UM3, pretreated flour, particle size is 150–250 μm.

pomace powders. The particle size reduction can alter or destroy the macromolecule matrix, thus resulting in some phenolic compounds being released or exposed, which was in agreement with this study [13]. Also, it has been noted that particle size reduction increases extraction yield because smaller particles decrease the solvent's path and speed up the process of extracting the sample's functional elements to their fullest extent [31].

Overall, contents and release rates of carotenoids in samples showed increasing trends with the decrease in particle size. This was suggested that particle size reduction of a matrix by crushing treatment could cause cell wall rupture, promoting carotenoids release into food products [50]. Additionally, the increased surface area of the flour particles can expose more of the outer layers of plant cells that contain carotenoids to the processing environment. Moreover, Speroni et al. [51] demonstrated that the use of micronization processing techniques increased the polyphenol content and antioxidant capacity of olive pomace. Zhang et al. [52] determined that fine grinding improved the accessibility of functional compounds in milled *Lycium ruthenicum* Murray, which increased their antioxidant abilities.

DPPH radical scavenging activity among pretreated and untreated pumpkin flours milled to different sizes was compared (Fig. 1). With the pretreatment and particle size reduction, the DPPH scavenging activities of flours rose. The 20UM1 flour showed the highest DPPH scavenging activity, followed by 20UM and 20UM2, while CON3 was the lowest compared to ascorbic acid scavenging activities. It was suggested that DPPH radical scavenging activity was highly related to the amount of total phenols, flavonoids, and carotenoids, which was supported by an investigation of Bai and Li [53]. The increase of antioxidant availability in the pretreated flours with smaller particle sizes (such as <75 and 100-75 μm) might be attributed to the fact that finer particles would be beneficial for the dissolution of free-form antioxidant compounds. Besides, particle size reduction broke the protein and fiber matrix structure and thus increased the availability of bound-form antioxidant compounds linked or embedded in the matrix [38].

3.4. Functional properties

The functional properties of pretreated and untreated pumpkin flour milled to different particle sizes are presented in Table 4 And it can be observed that pretreatment and particle size reduction improve the functional properties of flour. Flour sample 20UM1 had the highest values for WAC (11.45 ± 0.58 g/g), WSI (28.53 ± 0.75 %), SC (6.72 ± 0.10 g/g), and OAC (2.23 ± 0.20 g/g), while CON3 had the lowest values for these functional properties (7.55 ± 0.57 g/g, 20.30 ± 0.67 %, 5.05 ± 0.95 g/g, and 1.62 ± 0.15 g/g), respectively. According to Shevkani et al. [54] the significant differences in functional properties are probable due to the variation in protein content, which reveals the ability of flour to absorb water. The highest WAC observed in the smallest particle size might be due to the higher fiber content in this sample and its larger surface area, which increases the binding site for water [55].

The differences may have also been caused by hydrophilic groups in the cellulose and hemicelluloses of the flour, which resulted in easy integration with water; finally, the value of WAC increased. The increased WSI with decreasing particle size may be attributed to the greater specific surface area, resulting in higher leaching of soluble starch-derived molecules dissolved in water during the WAC assay [25].

The OAC values ranged from 2.23 ± 0.10 to 1.62 ± 0.15 g/g. The oil absorption capacity of flour increased with pre-drying treatment and particle size reduction, similar to the findings of (Ahmed et al. [27] in lentil flour and Benítez et al. [56] in onion fiber concentrate. According to Hitayezu and Kang [31], OAC is linked with the presence of hydrophobic protein, which is helpful in

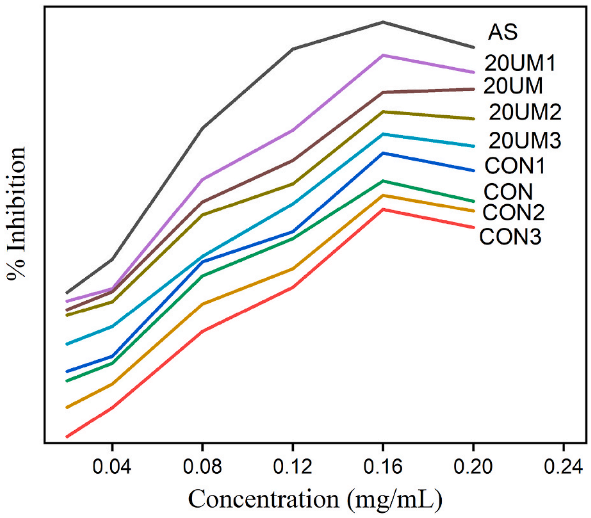

Fig. 1. Free radical scavenging activities of methanolic extract of pumpkin flour and ascorbic acid
AS, Ascorbic acid, as control; CON1, control flour, particle size is < 75 μm; CON, control flour, particle size is 75–100 μm; CON2, control flour, particle size is 100–150 μm; CON3, control flour, particle size is 150–250 μm; 20UM1, pretreated flour, particle size is < 75 μm; 20UM, pretreated flour, particle size is 75–100 μm and; 20UM2, pretreated flour, particle size is 100–150 μm; 20UM3, pretreated flour, particle size is 150–250 μm.

Table 4
Effects of interaction between particle size and pretreatment on functional properties of pumpkin flour.

Sample code	WAC(g/g)	WSI %	SC (g/g)	OAC (g/g)
CON1	9.34 ± 0.60^{bcd}	24.71 ± 0.79^{abc}	5.90 ± 0.53^{ab}	2.07 ± 0.20^{ab}
CON	8.56 ± 0.45^{cde}	23.18 ± 0.48^{abc}	5.63 ± 0.15^{ab}	1.95 ± 0.14^{ab}
CON2	7.93 ± 0.32^{de}	21.85 ± 1.43^{bc}	5.62 ± 0.23^{ab}	1.74 ± 0.17^{ab}
CON3	7.55 ± 0.57^{e}	20.30 ± 0.67^{c}	5.05 ± 0.95^{b}	1.62 ± 0.15^{b}
20UM1	11.45 ± 0.58^{a}	28.53 ± 0.75^{a}	6.72 ± 0.10^{a}	2.23 ± 0.20^{a}
20UM	10.89 ± 0.51^{ab}	27.30 ± 5.73^{ab}	6.13 ± 0.42^{ab}	2.18 ± 0.33^{a}
20UM2	10.34 ± 0.72^{ab}	25.89 ± 0.60^{abc}	5.97 ± 0.64^{ab}	2.12 ± 0.19^{ab}
20UM3	9.85 ± 0.714^{abc}	23.77 ± 0.97^{abc}	5.88 ± 0.67^{ab}	2.05 ± 0.09^{ab}
Effect				
Pretreatment (p)	***	***	***	***
Particle size (pz)	***	***	***	***
P × pz	***	***	***	NS

Values are mean ± standard deviation. Means sharing the same letters in columns are not significantly different from each other (Tukey's HSD test, $p < 0.05$). *** Significant effect at $p < 0.05$, NS, Not significant at $p < 0.05$. CON1, control flour, particle size is < 75 μm; CON, control flour, particle size is 75–100 μm; CON2, control flour, particle size is 100–150 μm; CON3, control flour, particle size is 150–250 μm; 20UM1, pretreated flour, particle size is < 75 μm; 20UM, pretreated flour, particle size is 75–100 μm and; 20UM2, pretreated flour, particle size is 100–150 μm; 20UM3, pretreated flour, particle size is 150–250 μm; WAC, water absorption capacity; WSI, water solubility index; SC, swelling Capacity; OAC, oil absorption capacity.

the binding of lipids. Swelling capacity is an important parameter that reflects the hydration ability. The increased surface area, polar groups, and other water-binding sites were exposed to the surrounding water medium, and the increased swelling capacities of 20UM1 and 20UM were potentially related to the flour particle size decreases [57]. This should be because the active sites in starch granules increased due to the structural destruction of flour under intense grinding treatment [58].

3.5. Pasting properties

The pasting properties of the flours obtained from pretreated and untreated pumpkin milled to different particle sizes are shown in Table 5; the viscosity increased with both pre-drying treatment and particle size reduction. As reported by Harasym, Satta and Kaim [59], ultrasound treatment partially disrupts starch granules and facilitates the formation of smaller and more numerous amylose and amylopectin molecules to increase viscosity during the heating process. Further, during microwave pre-drying treatment, the viscosity profile is modified due to the solubilization of the cell wall, causing disorganization and rupture of cellulose, fibre, and pectin, thus forming a firmer gel due to the higher content of linear chains that are responsible for gelatinization [60].

The peak viscosity indicates the starch's ability to swell before breakdown freely. The lowest peak and trough viscosity were recorded for the CON (660.04 ± 9.49cp, 637.92 ± 23.58 cp) flour sample, while the highest results were observed for the 20UM1 (780.40 ± 36.32, 687.76 ± 20.64 cp) flour sample, respectively. This could be attributed to the high crude fibre content (14.39 %) in 20UM1 flour, which is similar to the case reported by Fila, Itam and Johnson [61] and Adebowale, Adeyemi and Oshodi [62] for watermelon and six mucuna species, respectively. Higher peak viscosity for a 20UM1 (pretreated finer particle) indicates a faster rate of water absorption, which leads to starch granule swelling [63]. The lower peak viscosity of the CON (untreated coarser particle)

Table 5
Effects of interaction between particle size and pretreatment on pasting properties of pumpkin flour.

Flour Sample	Parameters						
	Peak viscosity (cp)	Trough (cp)	Breakdown (cp)	Final viscosity (cp)	Setback (cp)	Peak time (Min)	Pasting Temp (°C)
CON	660.4 ± 9.49^{b}	629.4 ± 19.21^{b}	31.0 ± 26.07^{a}	780.08 ± 10.32^{d}	150.68 ± 26.93^{a}	6.77 ± 0.06^{a}	00.00^{a}
CON1	684.44 ± 3.56^{b}	637.92 ± 23.58^{ab}	46.52 ± 27.09^{a}	817.56 ± 12.73^{c}	179.64 ± 36.18^{a}	6.02 ± 0.11^{b}	00.00^{a}
20UM	721.32 ± 39.62^{ab}	653.52 ± 23.88^{ab}	67.80 ± 63.29^{a}	856.00 ± 8.53^{b}	202.48 ± 31.23^{a}	5.64 ± 0.03^{c}	00.00^{a}
20UM1	780.40 ± 36.32^{a}	687.76 ± 20.64^{a}	92.64 ± 16.32^{a}	917.32 ± 16.78^{a}	229.56 ± 36.65^{a}	5.17 ± 0.02^{d}	00.00^{a}
Effect							
Pretreatment (p)	***	***	NS	***	***	***	NS
Particle size (pz)	***	***	NS	***	***	***	NS
Interaction (p × pz)	***	***	NS	***	NS	***	NS

Values are mean ± SD. Means that sharing the same superscript letters in columns is not significantly different from each other (Tukey's HSD test, $p < 0.05$). *** Significant effect at $p < 0.05$, NS, Not significant. CON, control flour, particle size is 75–100 μm; CON1, control flour, particle size is < 75 μm; 20UM, pretreated flour, particle size is 75–100 μm, and 20UM1, pretreated flour, particle size is < 75 μm; cp, centipoise.

sample could be due to lesser WAC [64]. Variation in peak viscosity between flours might be attributed to variation in amylose content, which would bring about variation in functional properties like starch crystallinity, amylose leaching, granule swelling, and also structural differences like branch chain length of amylopectin [65,66]. The peak viscosity values are significantly lower than that reported by (Promsakha na Sakon Nakhon et al. [67] and Ahmed et al. [27] for pumpkin flour (340–368 BU) and Indian lentil flour (544–714 BU), which increases with decreasing particle size, respectively.

The breakdown viscosity values of 20UM1, 20UM, CON1, and CON flour samples were 92.64 ± 16.32, 67.80 ± 63.29, 46.52 ± 27.09, and 31.00 ± 26.07 cp, respectively. The results showed that the breakdown viscosity value for the pretreated fine flour was higher than the other flours, indicating that it was less resistant to breakdown. On the other hand, the coarser flours exhibited good paste stability and strong shearing resistance, as indicated by their lower breakdown viscosity values [27].

However, no significant difference was observed in the breakdown viscosities between all flour samples. In line with this study Ghafoor et al. [68] also reported an increase in breakdown viscosity (625 %) for sonicated navy bean flour. According to (Harasym, Satta and Kaim [59], the degree of breakdown viscosity might also depend on other viscosity contributors such as proteins and fibres. The highest value of setback viscosity was recorded for the 20UM1 (229.56 ± 36.65 cp) flour sample, while the lowest was for CON (150.68 ± 26.93 cp) flour sample. As revealed in Table 5, setback viscosity increases with both pre-drying treatment and particle size reduction. Similar patterns were recorded by Ghafoor et al. [68] and Zhu and Li [69] in the sonicated navy bean flour and quinoa flour, respectively. The results indicated that smaller particle size flour had a higher setback viscosity, which suggested a greater tendency for retrogradation. This can be attributed to the larger surface area and exposure of starch granules to moisture during processing, which resulted in a higher degree of swelling and more rigid structure during cooling. These findings are consistent with previous studies bySandhu and Singh [70] on shallot flour and Khushbu et al. [64] on corn starch. The lower final viscosity value is obtained for the CON flour sample; comparatively did not easily form a viscous paste. An increase in the final viscosity of the 20UM1 sample might be due to a greater aggregation of amylose molecules in samples, which forms paste after heating and cooling [64]. There are significant differences ($p < 0.05$) in the peak time between both pretreated and control samples, with pretreated fine milled flour, which took a shorter time (5.17 min) to gelatinize, meaning that the control sample was slower and required more heating to form a paste. No pasting temperature was observed in both pretreated and untreated flours, similar to (Adelerin, Ifesan and Awolu [43] finding for pumpkin flour. It may be due to the starch granules begin to absorb water and swell gradually over a range of temperatures, rather than at a specific temperature. This was attributed to the presence of fibres and other non-starch components in the flour, which can affect the pasting properties and mask the pasting temperature [71].

3.6. Thermal properties

Table 6 presents the thermal properties of pumpkin flour; both temperature and enthalpy are affected by pre-drying treatment and particle size reduction. The findings indicated that applying a pre-drying treatment caused decreases in both temperature and enthalpy. On the other hand, reducing the particle size resulted in a temperature decrease (Table 6). While the CON flour sample showed the highest onset gelatinization temperature (69.70 ± 0.54 °C), peak gelatinization temperature (73.74 ± 0.85 °C), and conclusion gelatinization temperature (77.89 ± 0.71 °C), but 20UM1 (untreated flour with fine particle) showed the lowest temperatures, 62.92 ± 0.42, 66.55 ± 0.73 and 70.31 ± 0.80 °C, respectively. According to Chen et al. [72] ultrasound treatment can cause the breakdown of starch granules into smaller particles, which increases the surface area of the starch and lowers the gelatinization temperature, requiring less energy to reach the gelatinization point. Furthermore, their research suggests that ultrasound pre-drying treatment can lead to a decrease in the enthalpy value of the starch, which is attributed to the disruption of the starch granule structure caused by the ultrasound treatment. This disruption leads to a decrease in the amount of ordered crystalline regions within the starch granules. In addition, due to the waves' absorption by the starch granules, the mechanical impacts of ultrasonic waves cause heat to be produced. Because of this, the temperature of the starch may rise, accelerating the gelatinization process and further disorganizing the starch granules. The microwave radiation can generate heat and boost the temperature by rapidly vibrating the water molecules in the starch. This could result in the starch granules swelling and gelatinizing, which would disrupt the granule structure [60].

Table 6
Effects of interaction between particle size and pretreatment on thermal properties of pumpkin flour.

Flour Sample	Parameters			
	To (°C)	Tp (°C)	Tc (°C)	DH (J g^{-1})
CON	69.70 ± .535^{a}	73.74 ± 0.85^{a}	77.89 ± 0.71^{a}	4.53 ± 0.05^{b}
CON1	67.12 ± 0.52^{b}	71.00 ± 0.80^{b}	75.00 ± 1.00^{b}	5.23 ± 0.25^{a}
20UM	65.34 ± 0.52^{c}	69.12 ± 0.85^{b}	73.03 ± 1.02^{b}	1.85 ± 0.33^{d}
20UM1	62.92 ± 0.42^{d}	66.55 ± 0.73^{c}	70.31 ± 0.80^{c}	2.85 ± 0.15^{c}
Effect				
Pretreatment (p)	***	***	***	***
Particle size (pz)	***	***	***	***
Interaction (p × pz)	***	***	***	***

Values are mean ± SD. Means that sharing the same superscript letters in columns are not significantly different from each other (Tukey's HSD test, $p < 0.05$). *** Significant effect at $p < 0.05$, NS, Not significant. CON, control flour, particle size is 75–100 μm; CON1, control flour, particle size is < 75 μm; 20UM, pretreated flour, particle size is 75–100 μm, and 20UM1, pretreated flour, particle size is < 75 μm. To, onset temperature; Tp, peak temperature; Tc, conclusion temperature; DH, gelatinization enthalpy.

The decrease in temperature was attributed to an increase in the surface area of the starch granules, which led to a greater degree of swelling and more rapid gelatinization. Smaller flour particle sizes provide a larger surface area for starch granules to interact with water during gelatinization. This results in quicker and more thorough gelatinization of starch, which in turn leads to a greater amount of energy being released during the process. Therefore, smaller flour particle sizes may need less energy input to reach gelatinization, which can result in a greater overall enthalpy [73].

This is comparable to the results of Majid, Dar and Nanda [28] and Raza et al. [74] for unsprouted onion powder and extruded chickpea powder, respectively. According to Khushbu et al. [64], it is clear from the results that the 20UM1 sample can be used in the food industry for its application as a thickener, with a low energy requirement for creating a bond in an aqueous solution upon heating.

3.7. Structural characteristics

FTIR: FTIR was used as a monitoring quality control because it rapidly screened and quantified chemical components in samples [75]. Fig. 2 shows the FTIR spectra peaks of the pretreated and untreated pumpkin flours with different particle sizes at around 3299, 2896, 1607, 1377, and 1025 cm^{-1}. The findings reveal that the flour's general spectral profile was similar, and no new chemical group bands were created as a result of pre-drying treatment and particle size reduction, demonstrating that the flour's primary structural characteristics were preserved. Interestingly, while the intensity of bands decreased with pretreatment, it was usually increased with the flour particle size reduction. The intensity of the FTIR bands decreased due to the disruption of the granule structure caused by both the heating of the starch granules from the microwave radiation, which led to their swelling and gelatinization [76], and the mechanical force generated by the ultrasonic vibrations, which caused the granules to break apart, contract, and the granule structure to become chaotic [77].

Because of the increased surface area of the flour particles, FTIR band intensity may rise with reduction in flour particle size. This enhanced contact between the flour particles and FTIR radiation due to the larger surface area may increase the radiation's absorption and, in turn, the strength of the FTIR bands [78]. The intensity of bands of fine milled untreated flour with a particle size of <75 μm (CON1) was higher than the other three (20UM1, 20UM, and CON) flours intensity. The bands at 3299 cm^{-1} can be attributed to –OH stretching vibrations from carbohydrates or other compounds such as carboxyl acid and ketone [75]. The frequency at 2896 cm^{-1} is related to CH and CH_2 stretching vibrations [75]. The absorption bands at 1610 cm^{-1} are due to bound water [79], while that at 1377 cm^{-1} originates from CH symmetric bending. The bands at 1025 cm-1 were assigned to esters of lipids [75].

XRD: X-ray diffraction patterns of pumpkin flour obtained after pre-drying treatment and size reduction are shown in Fig. 3. The CON, CON1, 20UM, and 20UM1 flour samples showed a characteristic peak at diffraction angles 2θ of 21.8°, 21.7°, 21.1°, and 21.1°, respectively, with no significantly different at $P < 0.05$ (Table 7), suggesting that the peak position of the flour after pre-drying treatment and ball milling were not changed significantly. This could indicate that the crystallinity structure was not significantly modified by the interaction effect of pre-drying treatments and particle size. The main difference was a slight reduction in the diffraction intensities, especially in the case of pretreated flour (20UM and 20UM1). The result was in agreement with the conclusion of FTIR.

The results were compared based on pretreatment and particle sizes of flour, and it was implied that while the CON1 (untreated fine milled <75 μm) flour had the highest crystallinity index (61.57 %), 20UM (pretreated coarse milled 75–100 μm) flour sample had lowest crystallinity index (45.21 %), which is similar to Zhao et al. [80] finding for superfine grinding of cellulose fiber structure. In line with this, while the peak intensity significantly decreases with pretreatment but it increases with particle size reduction (Fig. 3). The starch granules break apart and became disordered as a result of the combined mechanical force and heat impacts produced by the ultrasonic and microwave treatments [81]. The average distance between particles size for CON, CON1, 20UM, and 20UM1were 23.2, 23.31, 24.00, and 24.00 nm, respectively with no significant difference ($P < 0.05$) between them. The decrease of crystallinity peaks of pretreated flours is due to disorganization in the structure of starch caused by the effect of ultrasound waves and microwave

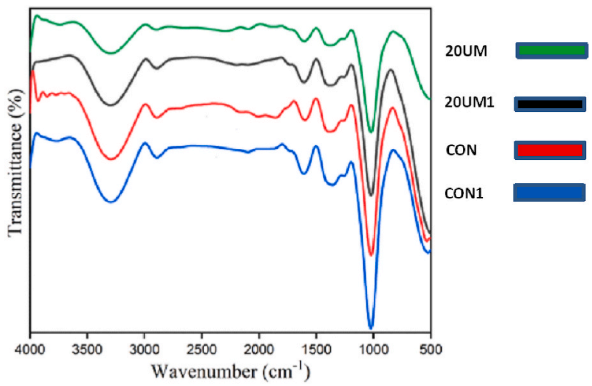

Fig. 2. Comparison of FTIR spectra of pumpkin flour
CON, control flour, particle size is 75–100 μm; CON1, control flour, particle size is < 75 μm; 20UM, pretreated flour, particle size is 75–100 μm and 20UM1, pretreated flour, particle size is < 75 μm.

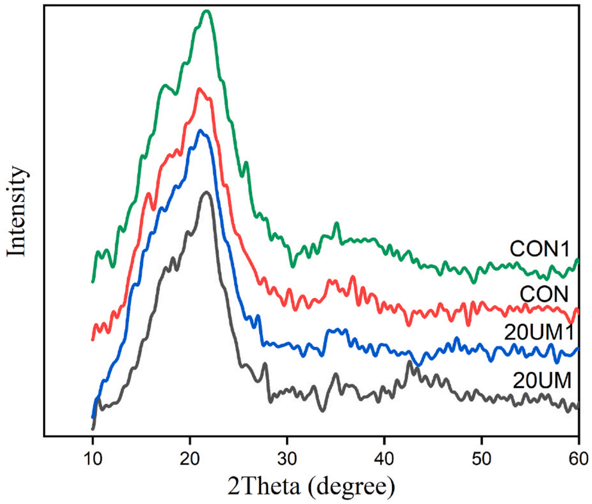

Fig. 3. XRD patterns of pumpkin flours
CON, control flour, particle size is 75–100 μm; CON1, control flour, particle size is < 75 μm; 20UM, pretreated flour, particle size is 75–100 μm; and 20UM1, pretreated flour, particle size is < 75 μm.

Table 7
Effects of interaction between particle size and pretreatment on XRD parameters of pumpkin flour.

Flour Sample	Parameters			
	Crystallinity index (%)	Peak diffraction (°)	Peak intensity	D-spacing (nm)
CON	54.99 ± 0.92^{b}	21.80 ± 0.42^{a}	319.00 ± 3.00^{b}	23.23 ± 0.40^{a}
CON1	61.58 ± 1.02^{a}	21.70 ± 0.55^{a}	360.00 ± 2.75^{a}	23.34 ± 0.25^{a}
20UM	45.22 ± 0.72^{d}	21.10 ± 0.50^{a}	282.00 ± 3.12^{d}	24.00 ± 0.05^{a}
20UM1	50.31 ± 1.12^{c}	21.10 ± 0.20^{a}	310.00 ± 3.05^{c}	24.00 ± 0.45^{a}
Effect				
Pretreatment (p)	***	NS	***	***
Particle size (pz)	***	NS	***	NS
Interaction (p × pz)	***	NS	***	NS

Values are mean ± SD. Means that sharing the same superscript letters in columns is not significantly different from each other (Tukey's HSD test, $p < 0.05$). *** Significant effect at $p < 0.05$, NS, Not significant. CON, control flour, particle size is 75–100 μm; CON1, control flour, particle size is < 75 μm; 20UM, pretreated flour, particle size is 75–100 μm; and 20UM1, pretreated flour, particle size is < 75 μm.

temperature. According to Trancoso-Reyes et al. [60], microwave heating generates greater movement of water molecules within the flour, producing major disorganization in the starch granules. Bashir and Aggarwal [82] found similar results, observing decreases in relative crystallinity linked to decreases in FTIR band intensities of irradiation chickpea starches. The lower H value indicates greater crystalline starch structure disorganization [83].

SEM: The surface morphology of pumpkin flour was observed using SEM at a magnification of 2000*, and the surface structure was changed with pre-drying treatment but not with particle size reduction (Fig. 4(a–d)). More swollen starch granules were observed for pretreated (20UM and 20UM1) flours, but agglomeration was observed on the untreated (CON and CON1) flours, which is in line with Trancoso-Reyes et al. [60] finding for 6min microwave pretreated sweet potato starch granules. Pre-drying treatments caused gelatinization and swelling of the starch granules, creating a more uniform and stable structure that prevented agglomeration. Contrarily, because intact starch granules were present in untreated fruit powders, they displayed microscale agglomeration [84]. There are also spaces between starch granules of pretreated flour (Fig. 4(a–d)). Untreated flour's agglomerated appearance may be due to its increased moisture content, which caused water to adsorb to the surface or become associated with fibre or other components [28]. The changed morphology can affect the physicochemical properties of the flour.

4. Conclusion

In conclusion, it has been shown that pre-drying treatment and particle size influence pumpkin flour's physical, chemical, functional, pasting, thermal, and structural properties. Pretreated (20UM1 and 20UM) and untreated (20UM1 and CON) pumpkin flour with various particle sizes had protein and crude fibre contents that ranging from 9.05 to 10.31 %, 6.99–8.00 %, and 12.4–14.39 %, 11.03–12.79 %, respectively. Size reduction increases particles' surface area and consequently improves the water and oil holding capacity and lightness of pumpkin flours. The study found that the pre-drying treatment and particle size reduction of the flours

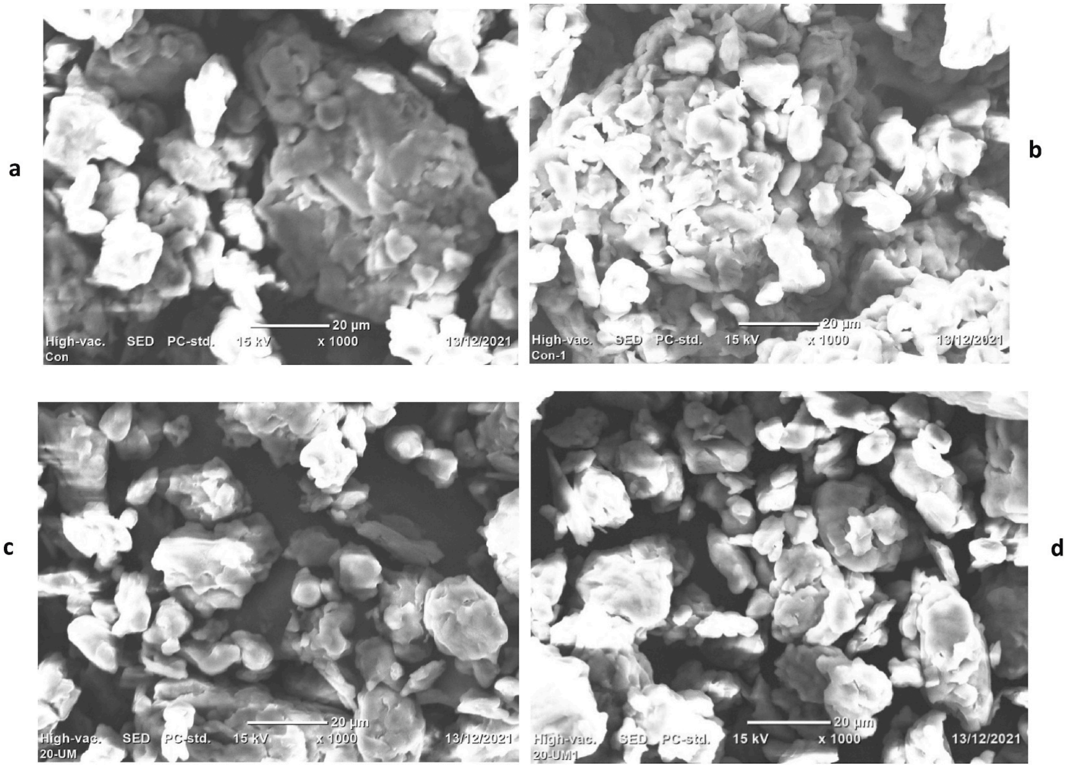

Fig. 4. SEM of pumpkin flours
a, CON, control flour, particle size is 75–100 μm; b, CON1, control flour, particle size is < 75 μm; c, 20UM, pretreated flour, particle size is 75–100 μm, and d, 20UM1, pretreated flour, particle size is < 75 μm.

increased the extraction of active compounds, potentially resulting in an improvement in the efficacy for scavenging DPPH radicals. Both pre-drying treatment and particle size reduction resulted in increasing pasting properties. Thermal temperature (To, Tp, and Tc) and change of enthalpy (ΔH) values decreased with pre-drying treatment and increase in particle size. The results of FTIR analysis showed that there was no change in the composition of the functional groups at the macromolecular level. X-ray analysis showed that the crystallinity index and intensity of prominent crystal peaks of fine milled untreated flour were the highest. Based on the examination of SEM images of pumpkin flour particles, the flour from the pre-drying treatment contained a swollen starch fragment. It can be concluded that sample 20UM1 is a superior component to increase the important ingredients in a variety of food formulations due to its better composition, thermal, functional, and structural characteristics when compared to others.

Data availability statement

Data included in article/supp. Material/referenced in article.

CRediT authorship contribution statement

Derese Wodajo Bekele: Conceptualization, Data curation, Formal analysis, Investigation, Methodology, Resources, Software, Writing – original draft, Writing – review & editing. **Shimelis Admassu Emire:** Conceptualization, Supervision, Writing – review & editing.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgments

Author Derese Wodajo would like to thank Wolkite University and Addis Ababa University Center of Food Science and Nutrition for providing laboratory facilities. Also, the authors appreciate the unspecified reviewers' watchful works and thoughtful suggestions.

Abbreviations

20UM	20min ultrasound followed by 6min microwaved pretreated flour with particle size 75–100 μm
20UM1	20min ultrasound followed by 6min microwaved pretreated flour with particle size <75 μm
20UM2	20min ultrasound followed by 6min microwaved pretreated flour with particle size <100–150 μm
20UM3	20min ultrasound followed by 6min microwaved pretreated flour with particle size 150–250 μm
CON	untreated pumpkin flour (control flour) with particle size 75–100 μm
CON1	untreated pumpkin flour (control flour) with particle size <75 μm
CON2	untreated pumpkin flour (control flour) with particle size 100–150 μm
CON3	untreated pumpkin flour (control flour) with particle size 150–250 μm

References

[1] M. Saeleaw, G. Schleining, Composition, Physicochemical and Morphological Characterization of Pumpkin Flour, 11th International Congresson Engineering and Food, 2011.
[2] M. Bhagya, G. Sanja, I. Marliya, EFFECT OF PRETREATMENT METHODS ON THE QUALITY OF DEHYDRATED PUMPKIN SLICES, 2018.
[3] F. Que, L. Mao, X. Fang, T. Wu, Comparison of hot air-drying and freeze-drying on the physicochemical properties and antioxidant activities of pumpkin (Cucurbita moschata Duch.) flours, Int. J. Food Sci. Technol. 43 (2008) 1195–1201.
[4] F. Hasturk Sahin, T. Aktas, H. Orak, P. Ulger, Influence of pretreatments and different drying methods on color parameters and lycopene content of dried tomato, Bulg. J. Agric. Sci. 17 (6) (2011) 867–881.
[5] T.S. Workneh, A. Zinash, K. Woldetsadik, Blanching, salting and sun drying of different pumpkin fruit slices, J. Food Sci. Technol. 51 (11) (2014) 3114–3123.
[6] Derese Wodajo, S.A. Emire, Pumpkin flour qualities as affected by ultrasound and microwave pre-drying treatment, Int. J. Food Prop. 25 (1) (2022) 2409–2424.
[7] M. Zhang, M. Zhang, B. Adhikari, R. Adhikari, Combined effect of ultrasound and microwave pretreatment on drying kinetics and quality of kiwifruit, J. Food Process. Preserv. 44 (5) (2020), e14454.
[8] G. Barbosa-Canovas, E. Ortega-Rivas, P. Juliano, H. Yan, Food Powders: Physical Properties, Processing, and Functionality, Kluwer Academic/Plenum Publishers, New York, 2005.
[9] Y. Lee, W. Yoon, Effects of particle size and heating time on thiobarbituric acid (TBA) test of soybean powder, Food Chem. 138 (2–3) (2013) 841–850.
[10] K.X. Zhu, S. Huang, W. Peng, H.F. Qian, H.M. Zhou, Effect of ultrafine grinding on hydration and antioxidant properties of wheat bran dietary fiber, Food Res. Int. 43 (4) (2010) 943–948.
[11] Shudong He, et al., Physicochemical and antioxidant properties of hard white winter wheat (Triticum aestivm L.) bran superfine powder produced by eccentric vibratory milling, Powder Technol. 325 (2018) 126–133.
[12] Q.M. Chen, M.R. Fu, F.L. Yue, Y.Y. Cheng, Effect of superfine grinding on physicochemical properties, antioxidant activity and phenolic content of red rice (*Oryza sativa* L.), Food Nutr. Sci. 6 (14) (2015) 1277–1284.
[13] F.M. Zhu, B. Du, B.J. Xu, 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. 65 (2015) 43–47.
[14] M. Siddiq, R. Ravi, J.B. Harte, K.D. Dolan, Physical and functional characteristics of selected dry bean (Phaseolus vulgaris L.) flours, LWT–Food Sci. Technol. 43 (2) (2010) 232–237.
[15] R.S. Reddy, C.T. Ramachandra, S. Hiregoudar, U. Nidoni, J. Ram, M. Kammar, Influence of processing conditions on functional and reconstitution properties of milk powder made from Osmanabadi goat milk by spray drying, Small Rumin. Res. 119 (1–3) (2014) 130–137.
[16] AOAC, Official Methods of Analysis. , Association of Official Analytical Chemist, 15th Editi. Washington D.C, 1990.
[17] AOAC, Association of Official Analytical Chemists, seventeenth ed., Washington DC, 2005.
[18] AOAC, Association of Official Analytical Chemists, 2006, eighteenth ed., Gaithersburg, 2006.
[19] A. of O. A. C. AOAC, Official Methods of Analysis of the Association of the AOAC, nineteenth ed., Association of Official Analytical Chemists Inc., Gaithersburg, 2012, p. 2012.
[20] Isabel Ferreira, B. Paula, M. Vilas-boas, L. Barros, Free-radical scavenging capacity and reducing power of wild edible mushrooms from northeast Portugal : individual cap and stipe activity, Food Chem. 100 (2007) 1511–1516.
[21] M. Minuye, P. Getachew, A. Laillou, S. Chitekwe, K. Baye, Effects of different drying methods and ascorbic acid pretreatment on carotenoids and polyphenols of papaya fruit in Ethiopia, Food Sci. Nutr. 9 (6) (2021) 3346–3353.
[22] A.Z. Woldegiorgis, D. Abate, G.D. Haki, G.R. Ziegler, Antioxidant property of edible mushrooms collected from Ethiopia, Food Chem. 157 (2014) 30–36.
[23] L.M.J. de Carvalho, et al., Total carotenoid content, α-carotene and β-carotene, of landrace pumpkins (Cucurbita moschata Duch): a preliminary study, Food Res. Int. 47 (2) (2012) 337–340.
[24] A.M. Goula, K.G. Adamopoulos, N.A. Kazakis, Influence of spray drying conditions on tomato powder properties, Dry. Technol. 22 (5) (2004) 1129–1151.
[25] X. Zhao, Z. Yang, G. Gai, Y. Yang, Effect of superfine grinding on properties of ginger powder, J. Food Eng. 91 (2) (2009) 217–222.
[26] Y. Fayin, T. Bingbing, J. Liu, Y. Zou, Effect of micronization on the physicochemical properties of insoluble dietary fiber from citrus (Citrus junos Sieb . ex Tanaka) pomace, Food Sci. Technol. Int. 0 (0) (2015) 1–10.
[27] J. Ahmed, A. Taher, M.Z. Mulla, A. Al-Hazza, G. Luciano, Effect of sieve particle size on functional, thermal, rheological and pasting properties of Indian and Turkish lentil flour, J. Food Eng. 186 (2016) 34–41.
[28] I. Majid, B.N. Dar, V. Nanda, Rheological, thermal, micro structural and functional properties of freeze dried onion powders as affected by sprouting, Food Biosci. 22 (January) (2018) 105–112.
[29] Y. Liu, L. Wang, F. Liu, S. Pan, Effect of grinding methods on structural, physicochemical, and functional properties of insoluble dietary fiber from orange peel, Int. J. Polym. Sci. (2016) 1–7.
[30] M.S. Hunter, et al., X-ray diffraction from membrane protein nanocrystals, Biophys. J. 100 (2011) 198–206.
[31] E. Hitayezu, Y. Kang, Effect of particle size on the physicochemical and morphological properties of Hypsizygus marmoreus mushroom powder and its hot-water extracts, Korean J. Food Preserv 28 (4) (2021) 540–549.
[32] Z. Wang, J. Huang, Y. Zhang, X. Li, G. Liu, Effects of particle size on the physicochemical and functional properties of rice flour, J. Cereal. Sci. 77 (2017) 74–80.
[33] Li Guanghui, W. Guo, X. Gao, Y. Wang, S. Sun, Effect of superfine grinding on physicochemical and antioxidant properties of soybean residue powder, Food Sci. Nutr. 00 (2020) 1–7.
[34] M.K. Bolade, I.A. Adeyemi, A.O. Ogunsua, Influence of particle size fractions on the physicochemical properties of maize flour and textural characteristics of a maize-based nonfermented food gel, Int. J. Food Sci. Technol. 44 (3) (2009) 646–655.
[35] J. Ahmed, H. Al-attar, Y.A. Arfat, Effect of Particle Size on Compositional, Functional, Pasting and Rheological Properties of Commercial Water Chestnut Flour, Food Hydrocoll., 2015.
[36] J. Ahmed, S. Al-Jassar, L. Thomas, A comparison in rheological, thermal, and structural properties between Indian Basmati and Egyptian Giza rice flour dispersions as influenced by particle size, Food Hydrocolloids 48 (2015) 72–83.

[37] A. Gani, S.S. Haq, F.A. Masoodi, A.A. Broadway, A. Gani, Physico-chemical, morphological and pasting properties of starches extracted from water chestnuts (trapa natans) from three lakes of kashmir, India, Braz. Arch. Biol. Technol. 53 (3) (2010) 731–740.
[38] S. Xianbao, et al., Effects of particle size on physicochemical and functional properties of superfine black kidney bean (Phaseolus vulgaris L.) powder, PeerJ 2019 (2) (2019).
[39] M.K. Mahawar, K. Jalgaonkar, V.E. Nambi, B. Bibwe, V. Thirupathi, Modelling of rheological properties of mango (cv. Neelum) suspensions: effect of sample concentration and particle size, J. Agric. Eng. 55 (4) (2019) 13–20.
[40] F. Fabiano A.N, L. Francisco E, R. Sueli, Ultrasound as pre-treatment for drying of pineapple, Ultrason. Sonochem. 15 (6) (2008) 1049–1054.
[41] B. Önal, G. Adiletta, M. Di Matteo, P. Russo, N. Ramos, C.L.M. Silva, Microwave and ultrasound pre-treatments for drying of the 'rocha' pear: impact on phytochemical parameters, color changes and drying kinetics, Foods 10 (853) (2021) 1–18.
[42] A.G. Farombi, A.M. Oyekanmi, Proximate , mineral and anti-nutrient evaluation of pumpkin pulp (cucurbita pepo), IOSR J. Appl. Chem. 4 (5) (2013) 25–28.
[43] R.O. Adelerin, B.O. Ifesan, O.O. Awolu, Physicochemical, nutritional, phytoconstituents, and antioxidant properties of three different processing techniques of pumpkin (*Cucurbita pepo*) pulp flour, Ceylon J. Sci. 51 (1) (2022) 43.
[44] J. Li, W. Zhang, X. Sun, Y. Liu, Effect of microwave pre-drying on the physicochemical properties of apple flour, J. Food Process. Preserv. 44 (9) (2020), e14614.
[45] O.O. Awolu, O.O. Adebo, A.S. Afolabi, Effect of particle size reduction on physicochemical properties of banana flour, J. Food Sci. Technol. 52 (4) (2015) 2261–2268.
[46] N. Boukhari, A. Doumandji, F.S.A. Chaouche, A. Ferradji, Effect of ultrasound treatment on protein content and functional properties of Spirulina powder grown in Algeria, Med. J. Nutrition Metab. 11 (3) (2018) 235–249.
[47] D.L. Luthria, K. Noel, D. Vinjamoori, Impact of sample preparation on the determination of crude fat content in corn, JAOCS, J. Am. Oil Chem. Soc. 81 (11) (2004) 999–1004.
[48] Z.H. Tekin, M. Baslar, The effect of ultrasound-assisted vacuum drying on the drying rate and quality of red peppers, J. Therm. Anal. Calorim. 132 (2) (2018) 1131–1143.
[49] X. Zhao, H. Zhu, G. Zhang, W. Tang, Effect of super fi ne grinding on the physicochemical properties and antioxidant activity of red grape pomace powders, Powder Technol. 286 (2015) 838–844.
[50] F. Lei, et al., Effect of particle size distribution on the carotenoids release, physicochemical properties and 3D printing characteristics of carrot pulp, Lwt 139 (2021), 110576.
[51] C.S. Speroni, et al., Micronization and granulometric fractionation improve polyphenol content and antioxidant capacity of olive pomace, Ind. Crops Prod. 137 (April) (2019) 347–355.
[52] J. Zhang, Y. Dong, T. Nisar, Z. Fang, Z.C. Wang, Y. Guo, Effect of superfine-grinding on the physicochemical and antioxidant properties of Lycium ruthenicum Murray powders, Powder Technol. 372 (2020) 68–75.
[53] Y.X. Bai, Y.F. Li, Preparation and characterization of crosslinked porous cellulose beads, Carbohydr. Polym. 64 (2006) 402–407.
[54] K. Shevkani, N. Singh, A. Kaur, J.C. Rana, Physicochemical, pasting, and functional properties of amaranth seed flours: effects of lipids removal, J. Food Sci. 79 (7) (2014).
[55] C.R. Köhn, A.M. Fontoura, A.P. Kempka, I.M. Demiate, E.H. Kubota, R.C. Prestes, Assessment of different methods for determining the capacity of water absorption of ingredients and additives used in the meat industry, Int. Food Res. J. 22 (1) (2015) 356–362.
[56] V. Benítez, E. Mollá, M.A. Martín-Cabrejas, Y. Aguilera, R.M. Esteban, Physicochemical properties and in vitro antidiabetic potential of fibre concentrates from onion by-products, J. Funct.Foods 36 (2017) 34–42.
[57] B. Du, F. Zhu, B. Xu, Physicochemical and antioxidant properties of dietary fibers from Qingke (hull-less barley) flour as affected by ultrafine grinding, Bioact. Carbohydrates Diet. Fibre 4 (2) (2014) 170–175.
[58] F. Hofmann, R.J. Harder, W. Liu, Y. Liu, I.K. Robinson, Y. Zayachuk, Glancing-incidence focussed ion beam milling: a coherent X-ray diffraction study of 3D nano-scale lattice strains and crystal defects, Acta Mater. 154 (2018) 113–123.
[59] J. Harasym, E. Satta, U. Kaim, Ultrasound treatment of buckwheat grains impacts important functional properties of resulting flour, Molecules 25 (13) (2020) 1–15.
[60] N. Trancoso-Reyes, L.A. Ochoa-Martínez, L.A. Bello-Pérez, J. Morales-Castro, R. Estévez-Santiago, B. Olmedilla-Alonso, Effect of pre-treatment on physicochemical and structural properties, and the bioaccessibility of β-carotene in sweet potato flour, Food Chem. 200 (2016) 199–205.
[61] W. Fila, E. Itam, J. Johnson, Comparative proximate compositions of watermelon (Citrullus Lanatus), squash (Cucurbita Pepo'l) and rambutan (Nephelium Lappaceum), Int. J. Sci. Technol. 2 (1) (2013).
[62] C.S. Martinez, P.D. Ribotta, A.E. León, M.C. Añón, PHYSICAL, sensory and chemical evaluation of cooked spaghetti, J. Texture Stud. 38 (2007) 666–683.
[63] S. Ragaee, Abdel-Aal, M. E.-S, Pasting properties of starch and protein in selected cereals and quality of their food products, Food Chem. 95 (1) (2006) 9–18.
[64] S. Khushbu, C.K. Sunil, D.V. Chidanand, R. Jaganmohan, Effect of particle size on compositional, structural, rheological, and thermal properties of shallot flour as a source of thickening agent, J. Food Process. Eng. 43 (3) (2020) 1–13.
[65] I.L. Batey, B.M. Curtin, Effects on pasting viscosity of starch and flour from different operating conditions for the rapid Visco analyser, Cereal Chem. 77 (6) (2000) 754–760, 77(6), 754–760.
[66] H.-J. Chung, Q. Liu, R. Hoover, T.D. Warkentin, B. Vandenberg, In vitro starch digestibility, expected glycemic index, and thermal and pasting properties of flours from pea, lentil and chickpea cultivars, Food Chem. 111 (2) (2008) 316–321.
[67] P. Promsakha na Sakon Nakhon, K. Jangchud, A. Jangchud, W. Prinyawiwatkul, Comparisons of physicochemical properties and antioxidant activities among pumpkin (Cucurbita moschata L.) flour and isolated starches from fresh pumpkin or flour, Int. J. Food Sci. Technol. 52 (11) (2017) 2436–2444.
[68] M. Ghafoor, N. Misra, K. Mahadevan, B. Tiwari, Ultrasound assisted hydration of navy beans (Phaseolus vulgaris), Ultrason. Sonochem. 21 (2014) 409–414.
[69] F. Zhu, H. Li, Modification of quinoa flour functionality using ultrasound, Ultrason. Sonochem. 52 (2019) 305–310.
[70] K.S. Sandhu, N. Singh, Some properties of corn starches II: physicochemical, gelatinization, retrogradation, pasting and gel textural properties, Food Chem. 101 (4) (2007) 1499–1507.
[71] S. Chaiwanichsiri, M. Suphantharika, P. Pinthusophon, Effects of flour particle size and added fibers on the pasting properties of cassava flour, J. Food Sci. Technol. 54 (11) (2017) 3509–3517.
[72] Y. Chen, B. Zhang, Z. Tong, J. Li, Effect of ultrasound treatment on the physicochemical properties of corn starch, Ultrason. Sonochem. 48 (2018) 426–433.
[73] G.S.V. Raghavan, L.R.V. Ramana, N.G. Malleshi, Effect of particle size on thermal properties of wheat flour, J. Food Sci. Technol. 52 (4) (2015) 2314–2319.
[74] R. Husnain, et al., Effects of ball-milling on physicochemical , thermal and functional properties of extruded chickpea (Cicer arietinum L.) powder, CyTA - J. Food 17 (1) (2019) 563–573.
[75] S.E. Quintana, R.M. Marsiglia, D. Machacon, E. Torregroza, L.A. Garcia-Zapateiro, Chemical composition and physicochemical properties of squash (Cucurbita moschata) cultivated in Bolivar department (Colombia), Contemp. Eng. Sci. 11 (21) (2018) 1003–1012.
[76] M. Zhang, H. Zhao, F. Xie, and L. Chen, "Effects of microwave treatment on the structure and physicochemical properties of potato starch.," Int. J. Biol. Macromol., vol. 167, pp. 1066–1074.
[77] M. Abdollahi, A. Farahnaky, M. Majzoobi, G. Mesbahi, Effect of ultrasound-assisted freeze-drying on the physicochemical properties of potato starch, Carbohydr. Polym. 15 (2016) 596–604.
[78] X. Wang, H. Sun, X. Li, Effect of particle size on the Fourier transform infrared spectra of wheat flour, J. Food Eng. 121 (2014) 129–133.
[79] J. Rojas, Y. Uribe, A. Zuluaga, Powder and compaction characteristics of pregelatinized starches, Pharmazie 67 (6) (2012) 513–517.
[80] H.B. Zhao, J.H. Kwak, Z.C. Zhang, H.M. Brown, B.W. Arey, J.E. Holladay, Studying cellulose fiber structure by SEM, XRD, NMR and acid hydrolysis, Carbohydr. Polym. 68 (2007) 235–241.
[81] J.O. Ugwuanyi, C.E. Ofoedu, R.U. Ofoegbu, Ultrasound-assisted microwave drying of cassava starch: effects on physicochemical properties and microstructure, J. Food Process. Eng. 41 (3) (2018), e12714.

[82] K. Bashir, M. Aggarwal, Physicochemical, thermal and functional properties of gamma irradiated chickpea starch, Int. J. Biol. Macromol. 97 (2017) 426–433.
[83] C. xia Li, Y. ying Liu, H. sheng Feng, S. zhen Ma, Effect of superfine grinding on the physicochemical properties of bulbs of Fritillaria unibracteata Hsiao et K.C. Hsia powder, Food Sci. Nutr. 7 (11) (2019) 3527–3537.
[84] S. Garg, P. Rawat, R.R.B. Singh, Effect of ultrasound and microwave pre-treatments on the physicochemical and functional properties of peach and pear powders, J. Food Eng. 261 (2019) 1–8.