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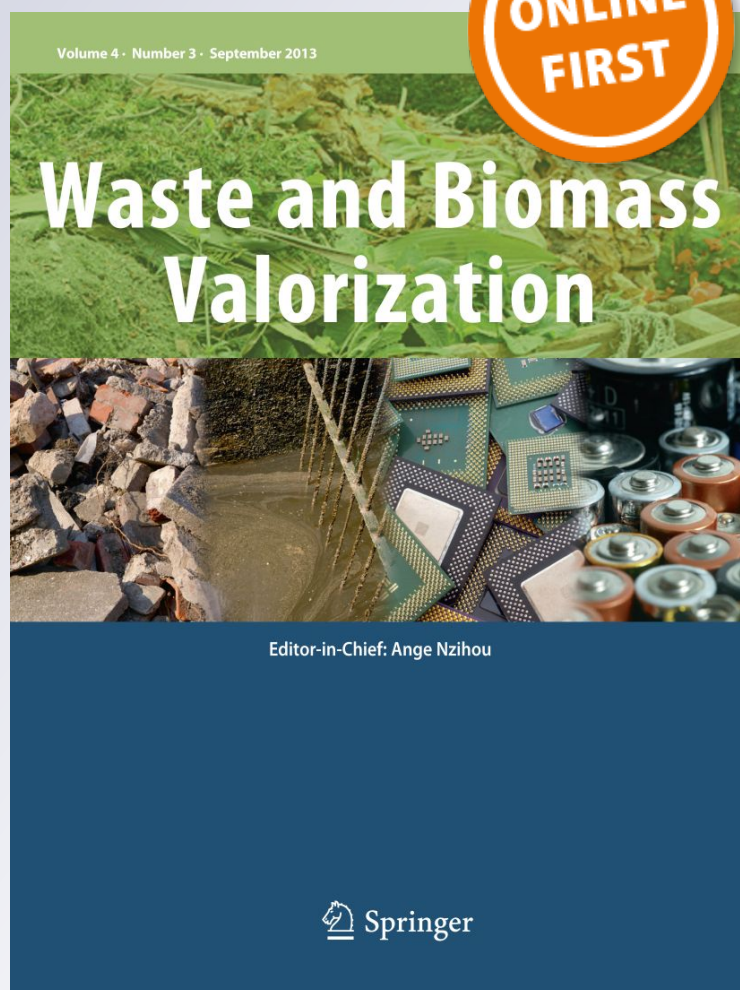
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Ultrafine Grinding a Promising Method for Improving the Total Dietary Fiber Content and Physico-Chemical Properties of Potato Peel Waste

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Abstract

This study aims at determining the microstructure, particle size, composition ratio and physicochemical properties of ultrafine pulverized potato peel waste (PPW) obtained from expanded and unexpanded extrusion methods. Results obtained in our study show that ultrafine grinding processes significantly modify the structural properties of PPW by significantly reducing the size and distribution of particles in the prepared powder. We have extensively determined the total dietary fiber (TDF) content and hydration characteristics, cholesterol, sodium cholate and fat absorption capacities of the extruded PPW. Results show that with a uniform increase in the grinding time, total dietary fiber content (especially the total proportion of the soluble dietary fiber) and hydration characteristics including water retention and swelling capacity (SWC) of extruded PPW were found to increase significantly. We have also observed that ambient pH and liquids modified the structural properties of extruded PPW to varying degrees. Interestingly, we have observed no significant difference between the PPW samples extruded by expanded and unexpanded processes, which eliminated the redundant extrusion step before the ultrafine grinding process. Thus, ultrafine grinding is a time-efficient, cost-effective and user-friendly method for processing PPW. Results obtained in this study endorse the great potential of PPW in the food and beverage industries. Our future studies will be focused on understanding the influence of extruded PPW in enhancing the total dietary fiber content, food texture, storage and taste of various commercial food products.

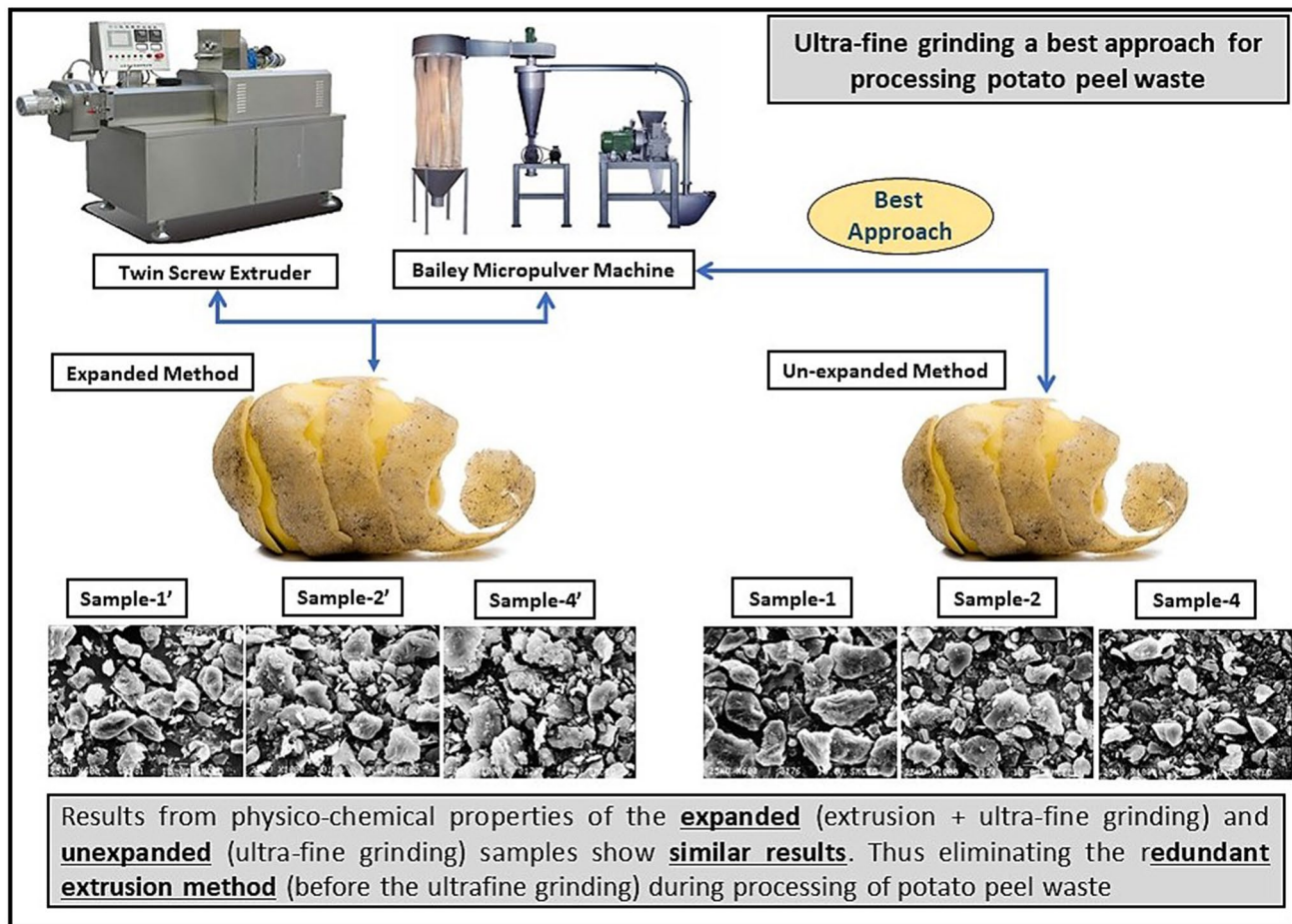
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Graphical Abstract



Keywords Potato peel · Dietary fiber · Extrusion · Ultrafine grinding · Particle size · Physicochemical properties

Abbreviations

PPW	Potato peel waste
TDF	Total dietary fiber
SDF	Soluble dietary fiber
IDF	Insoluble dietary fiber
WHC	Water holding capacity
SWC	Swelling capacity
NSAC	Unsaturated fatty acid absorption capacity
SAC	Saturated fatty acid absorption capacity
CAC	Cholesterol absorption capacity
SCAC	Sodium chelate absorption capacity
Expanded	Extrusion + ultrafine grinding
Unexpanded	Only ultrafine grinding

Statement of Novelty

A novel ultrafine grinding method was developed for processing the PPW obtained as a food-industry residue. For the first time, we have developed and compared the physico-chemical properties of PPW granules obtained from extrusion and extrusion-ultrafine, grinding-based processing methods. Our study reports that an ultrafine grinding-based method can be used for efficient processing of PPW by eliminating prior extrusion methods. We have also reported that PPW can potentially be used as a dietary fiber-rich food supplement for enhancing the physico-chemical properties of commercial food products.

Introduction

Dietary fiber is one of the major constituents of the human diet. Several studies have extensively reported about the positive impact of dietary fiber on human health [1, 2]. Usage and significance of dietary fiber in the human diet has resulted in the more recent development of a huge potential market for fiber-rich food products. Fiber-rich and fiber-supplemented food products are increasingly gaining popularity in the food industries [3, 4]. Dietary fiber is mainly obtained from plant material and is classified into soluble and insoluble dietary fibers. The soluble dietary fibers include pectin, gums and mucilage's, whereas insoluble dietary fiber includes plant cell wall poly and oligosaccharides (cellulose, hemicellulose) and non-carbohydrate lignin components [5, 6]. The complex chemical composition of dietary fiber exhibits various functions and activities as it passes through the gastrointestinal tract, and these functions and activities are strongly dependent on the physicochemical properties of fiber [6].

Potato granule powder has been widely used in the food industries. Especially during the preparation of potato

puree, potato chips, puffed foods, baked products, salads and other food products. Industrially grown potatoes are processed by peeling the skin, which produces between 15–40% of industrial food waste depending on the type of peeling process used [1, 7, 8]. Currently, only about 3% of the potato skin byproducts produced by the backfill method during granule production are used for cattle feed.

Based on a dry basis, the physicochemical properties of PPW exhibits about 15% of crude protein, 2.3% of crude fat, 3.3% of ash, 33.7% of crude starch, and 45.1% of TDF. It is one of the richest sources of dietary fiber thus, potentially exhibits higher industrial applications. PPW is an inexpensive source of feedstock for ruminating animals as it contains good quantities of starch, non-starchy polysaccharides, phenolic compounds such as lignin polyphenols, proteins and small quantities of lipids respectively. However, the high fibrous nature of PPW makes it unsuitable as feed for the non-ruminants, and it can be used after a pre-treatment [7, 9]. PPW can act as a valuable substrate for the extraction of antioxidants, dietary fiber, biopolymers and other valuable products [10–12]. Several studies in the past have revealed extensively about the compositions of TDF, SDF and IDF in various food sources (Fig. 1) [6, 13–15].

Fig. 1 Representation of dietary fiber content, total dietary fiber (Total DF), insoluble dietary fiber (IDF) and soluble dietary fiber (SDF) of various food materials (fruits, vegetables, nuts and seeds, grains, legumes and pulses) represented in g/100 g of edible portion. (Note: The above information was retrieved from ref. [6])

Grains	Total D.F	I.D.F	S.D.F
Barley	17.3	–	–
Corn	13.4	–	–
Oats	10.3	6.5	3.8
Rice (dry)	1.3	1	0.3
Rice (cooked)	0.7	0.7	0
Wheat (Whole grain)	12.6	10.2	2.3
Wheat germ	14	12.9	1.1

Legumes & pulses	Total D.F	I.D.F	S.D.F
Green beans	1.9	1.4	0.5
Soy	15	–	–
Peas, green frozen	3.5	3.2	0.3
Kidney beans, canned	6.3	4.7	1.6
Lentils, raw	11.4	10.3	1.1
Lima beans, canned	4.2	3.8	0.4
White beans, raw	17.7	13.4	4.3

Nuts and Seeds	Total D.F	I.D.F	S.D.F
Almonds	11.2	10.1	1.1
Coconut, raw	9	8.5	0.5
Peanut, dry roasted	8	7.5	0.5
Cashew, oil roasted	6	–	–
Seasame seed	7.79	5.89	1.9
Flaxseed	22.33	10.15	12.18

Vegetables	Total D.F	I.D.F	S.D.F
Potato, no skin	1.3	1	0.3
Bitter gourd	16.6	13.5	3.1
Beetroot	7.8	5.4	2.4
Fenugreek leaves	4.9	4.2	0.7
Ladyfinger	4.3	3	1.3
Spinach, raw	2.6	2.1	0.5
Turnips	2	1.5	0.5
Tomato, raw	1.2	0.8	0.4
Green onions, raw	2.2	2.2	0
Eggplant	6.6	5.3	1.3
Cucumbers, peeled	0.6	0.5	0.1
Cauliflower, raw	1.8	1.1	0.7
Celery, raw	1.5	1	0.5
Carrot, raw	2.5	2.3	0.2
Broccoli, raw	3.29	3	0.29

Fruits	Total D.F	I.D.F	S.D.F
Apple,unpeeled	2	1.8	0.2
Kiwi	3.39	2.61	0.8
Mango	1.8	1.06	0.74
Pineapple	1.2	1.1	0.1
Pomegranate	0.6	0.49	0.11
Watermelon	0.5	0.3	0.2
Grapes	1.2	0.7	0.5
Oranges	1.8	0.7	1.1
Plums	1.6	0.7	0.9
Strawberry	2.2	1.3	0.9
Bananas	1.7	1.2	0.5
Peach	1.9	1	0.9
Pear	3	2	1

Previous studies have reported that raw PPW exhibits higher carbohydrates, moisture contents, lower protein and lipid contents (water: 84.2%, protein: 1.8%, total lipids: 0.3%, total carbohydrates: 10.06%, starch: 7.8%, TDF: 2.5%, and ash: 1.3%) [7, 10, 16]. TDF is an important component of functional foods with unique physiological effects. The daily nutritional requirement for TDFs can potentially be enhanced by using PPW as a food supplement. Studies have reported that TDF significantly improves the physico-chemical properties of food materials (e.g., bread) [2]. In nature, most TDF occurs as water insoluble dietary fiber (IDF). The physico-chemical properties and physiological functions of IDF are closely related to its physical properties such as WHC and expansion rate [3]. However, the physical properties of TDF are further related to its chemical structure and its multi-phase intricate network. There are amorphous and crystalline regions in the network structure which can be differentiated into hydrophilic and hydrophobic regions. Maintenance of this intricate network structure depends internally on its chemical bonds, which in turn affect its physico-chemical properties. Therefore, modification of TDF has received greater attention around the world [4]. Earlier studies have reported on various TDF modification methods including chemical, biological, and physical methods, mainly ultra-micro-pulverization, ultra-high pressure, freeze-pulverization, extrusion cooking, and nanotechnology-based techniques were applied on various food sources including soybean hull [2], soybean [4] and bean residue [5, 6], sweet potato [7], wheat bran [8], and rice bran [9]. Results obtained from these studies reported that sample size, composition, physico-chemical properties and other aspects change considerably after processing. Comprehensive articles focused on the background, advantages and disadvantages of various extrusion techniques employed in the food industry (especially ultrafine super-pulverization) have also been reported previously [17–24].

Such studies were being conducted to explore the potato starch produced from the potato peel waste (a food-industry residue). Arapoglou et al., reported the composition and properties of potato peel residue for the first time [10]. The aim of the present study was to determine the microstructure, particle size, composition ratio and physico-chemical properties of ultrafine pulverized PPW obtained from expanded and unexpanded extrusion methods. Also, we have extensively reported the physico-chemical properties of the PPW obtained from the potato-based food industry. The PPW was used as the raw material to understand the effects of extrusion and ultrafine grinding technology on its particle distribution, TDF composition ratio, and other physico-chemical properties respectively. Our study reported that only the ultrafine grinding method can generate industrial grade food supplements with efficient physico-chemical properties. Various beneficial properties of PPW and

its great potential as an industrial food supplement and its potential ability to enhance the physico-chemical properties of commercial food products are the driving forces of our future studies [25].

Materials and Methods

Sample Preparation

The PPW used in our experiment was kindly provided by Shanxi Sanlai Food Co., Ltd. The PPW was obtained as a by-product during production of potato granule powder. Industrially, the potato granule powder is prepared by washing, peeling, slicing, rinsing, blanching, cooking, mashing, stirring, drying and sieving processes. The PPW powder appears in light, yellow-colored coarse particles with a particle size of about 0.3 cm and a moisture content less than 10%. We have used the standard dietary fiber test kit obtained from the Merk KGaA®. The peanut oil and chicken eggs used in our experiment were purchased from a local market in Thunder Bay, Ontario. Analytical grade cholesterol and sodium cholate reagents were purchased from the China National Pharmaceutical Group Chemical Testing Co., Ltd. All the biochemical reagents and materials used in our study were of analytical grade. We have summarized the step-by-step workflow implemented in our experiment in Fig. 2.

Extrusion Treatment

The PPW was extruded using a DS32-II Twin Screw Extruder (Jinan Saixin Machinery Co., Ltd) with four electrically heated and water-cooled extruder modules. The PPW used in our experiments was primarily measured for its moisture content. The PPW was subjected to a single extrusion procedure with a gradient increase in temperatures from 90, 120 and 170 °C, with the screw speed and PPW input rate maintained constantly at 30 Hz and 20 kg/h respectively. The PPW was subjected to the extrusion treatment until we achieved a specific dry weight of 1.162 mL g⁻¹ [26].

Ultrafine Grinding

The BFMT-6BI Bailey Micropulver Machine (Jinan Beili Powder Technology Engineering Co., Ltd, China) was used for performing ultrafine grinding of 2 kg PPW. Prior to ultrafine grinding, the PPW was dried at 75 °C in a hot air oven to remove the excess moisture content, and the process of drying was continued until we achieved a constant mass. The grinding speed of the micropulver machine was maintained constantly at 12,000 r/min, and the efficiency of grinding was determined by performing the grinding process

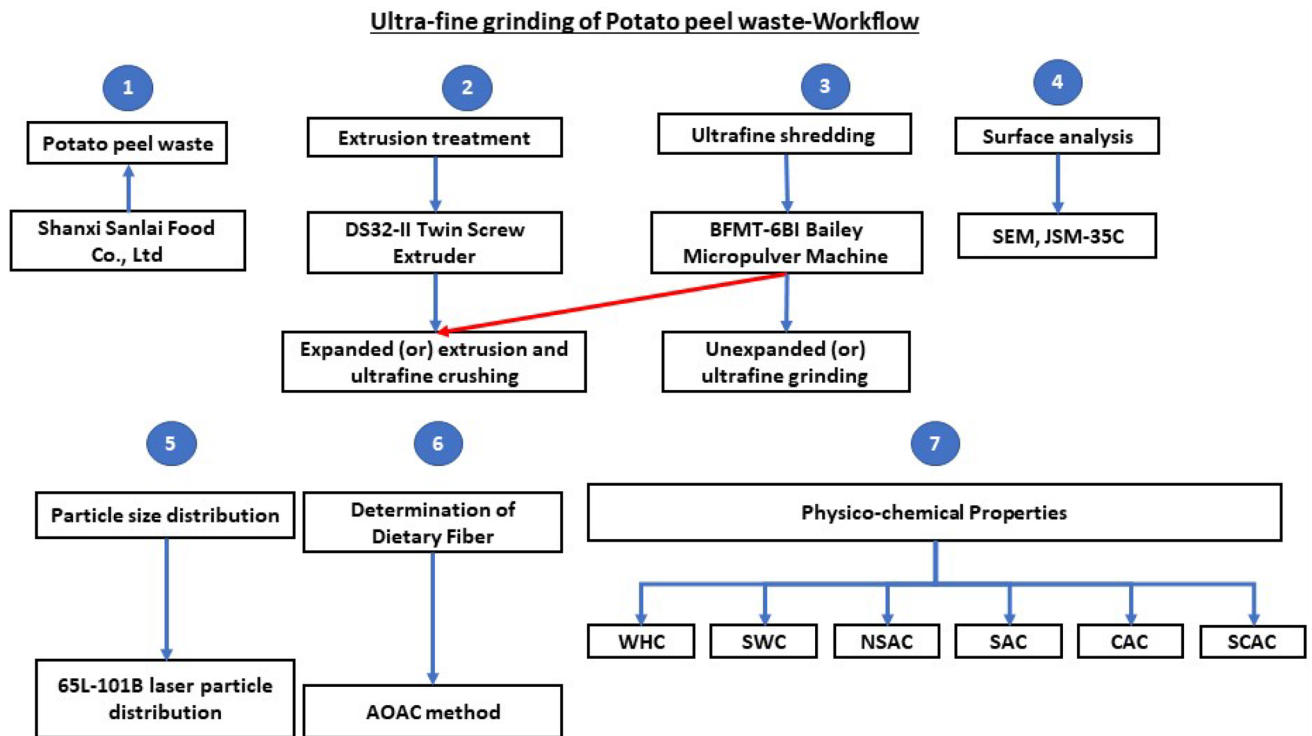


Fig. 2 Pictorial representation of experimental workflow used for studying the physico-chemical properties of potato peel waste (PPW) samples using the expanded and unexpanded extrusion methods

at various time periods ranging from 1, 5, 8, 13, 18 and 25 min respectively. A total of 12 samples were subjected to ultrafine grinding which resulted in fine powders of different sizes. These samples were differentiated and marked as unexpanded to refer to the ultrafine grinding process (sample 1–6), whereas those marked expanded referred to samples subjected to extrusion followed by an ultrafine grinding process (sample 1'–6').

SEM-Based Analysis

The analysis of the ultrafine shredded samples used the JSM-35C scanning electron microscope (Japan Electronics Co. Ltd, Japan). The ultrafine shredded PPW samples were adhered to the copper stage using double-sided tape and this section was sprayed with gold in a low-pressure argon atmosphere with a sputter coater at an accelerating voltage of 25 kV. These sputter coated samples were observed and photographed under the scanning electron microscope.

Particle Size Distribution

The particle size distribution of the extruded PPW residues was determined using a 65L-101B laser particle distribution measuring instrument (Liaoning Instrument Research Institute Co. Ltd, China) using the standard conditions. The

instrument was equipped with a sample presentation unit and a refractive index of 1.590 (Rosell et al. 2009). All the experiments were conducted in triplicate with 1 g of PPW sample dispersed using the ultrasonicator (95% ethanol used as a dispersant) for determining the hydrated particle size distribution of the PPW samples. Size distribution was quantified as the relative volume of particles in size bands presented as size distribution curves (Malvern Mastersizer Micro software v 5.40). Particle size distribution parameters recorded included the specific surface area with a largest particle size of (D90) [27].

Determination of Dietary Fiber

Dietary fiber present in the ultrafine shredded PPW was determined using the standard conditions of the AOAC method [27–30]. Initially, the samples were treated with thermo-stable α -amylase, amyloglucosidase and later it was digested with proteases obtained from P3910, Merk KGaA® to remove starch and protein from the samples. The insoluble dietary fiber content of the samples was then analyzed by centrifuging (LXT-HB Multi-tube Centrifuge Shanghai Jinpeng Analytical Instrument Co. Ltd, China) the samples post enzymatic treatment. The samples were concentrated by using a RE-52A Rotary Evaporator (Shanghai Arong Biochemical Instrument Co. Ltd, China). After determining

the IDF, samples were analyzed for the soluble dietary fiber by precipitating with 95% ethanol using a SHB-3 Vacuum Decompression Thickener (Zhengzhou Rhododendron Instrument Factory, China). Finally, the TDF was determined by adding the obtained IDF and SDF values.

Physio-Chemical Properties

The physicochemical properties of both the unexpanded and expanded PPW samples were determined, i.e., water holding capacity (WHC), swelling capacity (SWC), unsaturated fatty acid absorption capacity (NSAC), saturated fatty acid absorption capacity (SAC), cholesterol (CAC), and sodium cholate absorption capacity (SCAC) [27].

- (a) *Water-holding capacity (WHC)* was determined by adding 20 mL of distilled water and 2.5 g of PPW to the 50 mL plastic centrifuge tubes. The sample tubes were mixed thoroughly and stored at room temperature. After 24 h of incubation at room temperature, the samples were centrifuged at $1500\times g$ for 15 min, then decanted to record the bed volume and expressed as WHC (g water/g powder) by calculating the total water mass by dry matter mass [27, 29, 31].
- (b) *Swelling capacity (SWC)* was determined by adding 20 mL of distilled water and 0.5 g of PPW powder to the 50 mL centrifuge tube. The sample tubes were mixed thoroughly and stored at room temperature. After 16 h of incubation at room temperature, the samples were centrifuged at $1500\times g$ for 15 min, then decanted to record the bed volume. SWC is expressed in milliliters per gram of sample [27, 29].
- (c) *Unsaturated fatty acids absorption capacity (NSAC)* was determined by adding 1.50 g (W1) of sample in a centrifuge tube, and then adding 12.0 g of edible peanut oil. After adding these reagents, we incubated the centrifuge tubes at 37 °C for 1 h followed by centrifugation at 4000 r/min for 20 min. After centrifugation, we removed the upper layer of oil using a filter paper followed by weighing the filtered peanut oil (W2). The oil absorption capacity of PPW samples was calculated using $(g/g) = (W2 - W1)/W1$ respectively [28].
- (d) *Saturated fatty acid absorption capacity (SAC)* was determined by adding 1.00 g (W1) of sample in a centrifuge tube, then adding 10.0 g of lecithin and incubating at 37 °C for 1 h, followed by centrifugation at 4000 r/min for 20 min. After centrifugation, we removed the upper layer of oil using a filter paper followed by weighing the filtered oil (W2). The oil absorption capacity of PPW samples was calculated using $(g/g) = (W2 - W1)/W1$ [28].
- (e) *Cholesterol absorption capacity (CAC)* was calculated using fresh egg yolk separated from each egg

and diluted with 9 parts of deionized water. We used two 100 mL Erlenmeyer flasks and added 1.0 g of test sample to each tube followed by 25.0 g of diluted egg yolk solution. We adjusted the pH of one test tube to pH 2.0 using 0.01 N HCl and one test tube to pH 7.0 using 0.01 N NaOH. Both the test tubes were incubated in a shaking water bath (HES-HA Water Bath Constant Temperature Oscillator, Harbin Dongming Medical Instrument Factory, China) at 37 °C, 200 rpm for 2 h. After incubation, the sample solutions in both the test tubes were adjusted to 50 mL with deionized water, followed by centrifugation at 4000 r/min for 20 min. The final reaction mixture contained 1.0 mL of supernatant diluted with 5 mL of 90% acetic acid, 1 mL phthalaldehyde and 4 mL of premixed acid solution (equal volume of concentrated sulfuric acid and 90% acetic acid). The test samples were mixed thoroughly, with the volume of the test tubes adjusted to 10 mL with 90% acetic acid, and after adding all the reagents, the samples were left at room temperature. After 10 min, we recorded the absorbance of the test samples at 550 nm using a spectrophotometer (Model 721E-Visible Light Splitter Photometer). The CAC was calculated using the standard curve. The diluted egg yolk solution without the test sample was used as control. (Standard curve preparation was to take standard cholesterol application solution of 0.1 mg mL^{-1} , putting 0 mL in the blank tube, and 0.1 mL, 0.2 mL, 0.3 mL, 0.4 mL in each of 4 other tubes, adding 1 mL into each of phthalaldehyde reagent, then adding the mixture. We have made 4 mL of acid solution with equal volumes of concentrated sulfuric acid and 90% acetic acid solutions are added to make up to 10 mL with 90% acetic acid, and are later allowed to stand at room temperature for 10 min. The 721-type spectrophotometer was used to calibrate the zero point with the blank tube. The absorbance was plotted on the ordinate and the cholesterol concentration plotted on the abscissa. The equation used is $y = 206.21x - 0.0355$ $R^2 = 0.9991$) [27, 28].

- (f) *Sodium cholate absorption capacity* was determined by adding 1.0 g of the test sample to the 200 mL Erlenmeyer flask, followed by adding the sodium cholate reaction mixture containing 100 mL of 0.1 mol/L phosphate buffer (pH 7) supplemented with 0.15 mol/L solution of NaCl (0.2 g) and sodium cholate (0.3 g). We gently mixed the sample solution and incubated it at 37 °C for 2 h and later centrifuged it at 4000 r/min for 20 min. The final reaction mixture contained 1 mL of the supernatant, 6 mL of 45% sulfuric acid and 1 mL of 0.3% furfural solution. Finally, the above reaction mixture was incubated at 65 °C for 30 min and then cooled down to room temperature, and we later recorded the change in absorbance of the test solu-

tion at 620 nm using a spectrophotometer. The concentration of the sodium cholate absorption was calculated using the standard curve. The phosphate buffer without the sample was considered as control. SCAC was analyzed by calculating the difference between the initial and final concentrations of the reaction. (Standard curve preparation was to accurately prepare 0.1 mg mL^{-1} , 2 mg mL^{-1} , 3 mg mL^{-1} , 4 mg mL^{-1} , and 5 mg mL^{-1} sodium cholate standard solutions (0.1 mol/L , pH 7.0 sodium phosphate buffer solution), then 1 mL of each was pipetted into 15 mL stoppered tubes. Then we added 6 mL of 45% sulfuric acid to each tube, mixed well, added 1 mL of 0.3% furfural solution, mixed again, set a constant temperature water bath at 65°C for 30 min, cooled to room temperature, at 620 nm. The absorbance was measured by plotting on the ordinate, and the sodium cholate concentration plotted on the abscissa. The equation used is $y = 0.2761x - 0.0175$, $R^2 = 0.9976$) [28].

Data Analysis

All the experiments were statistically designed and were conducted in quadruplicates respectively. The results

obtained from all the test samples were analyzed to find the mean, \pm standard error ($\bar{x} \pm \text{SE}$). We have used SPSS 18.0® statistical software to process the data, and the significance of these results were analyzed using Duncan's new complex-difference and T test methods respectively with a p -value < 0.05 .

Results and Discussion

Effect of Extrusion and Ultrafine Grinding of PPW

In our current study, we have performed a combination of ultrafine grinding and extrusion crushing based methods for processing PPW samples. The ultrafine grinding treatment of PPW resulted in a fine powder with a large particle size and a compact structure. Results obtained from the SEM analysis of both the expanded and unexpanded PPW samples are reported in Fig. 3. Initially, we analyzed the moisture content of the selected PPW samples and it was found to be 35%, whereas the moisture content of pulverized PPW samples was found to be 12%. Earlier studies have reported that extrusion treatments performed under high temperature, pressure and shear force significantly breaks various

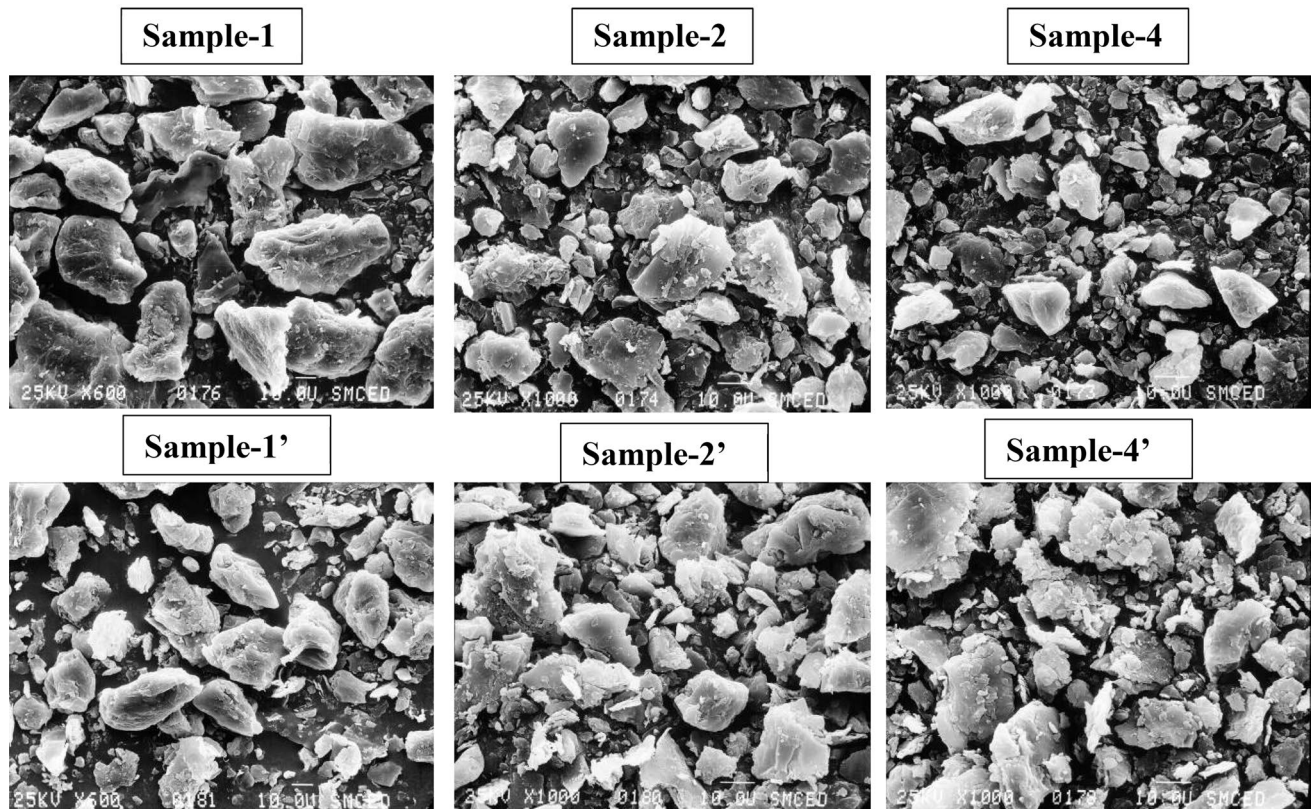


Fig. 3 Ultrafine extrusion of potato peel samples using samples 1, 2 and 4 (unexpanded) and samples 1', 2' and 4' (expanded) obtained from the extrusion processes

chemical bonds of the structural components [32–34]. Thus, altering the natural crystal structure to a highly disordered form potentially results in scaly and loose pores particles. We have also observed that with increases in degree of refinement, the larger sized particles were reduced to small-sized particles. Although, we have not conducted experiments related to surface area of the extruded PPW, our results suggest that particle size is inversely proportional to the surface area and similarly, as the particle size decreases, we might observe an increase in surface area. Finally, our results strongly endorse the previous studies which suggested that, extrusion treatments might potentially alter the surface of the PPW to varying degrees. However, further studies need to be conducted in this regard [32–34] (Fig. 3).

The significance of PPW as a potential source of dietary fiber is increasingly gaining attention in the food industries [32]. Previous studies have reported that the process of extrusion cooking of PPW significantly changes its functional and structural properties. In our study, we have observed that extruded PPW exhibits two commercially important features: color and TDF content [32, 33]. Studies have shown that both extrusion and ultrafine grinding techniques would significantly alter the physico-chemical properties of PPW [6, 8]. According to Camire et al., extrusion of PPW resulted in increasing concentrations of uronic acids in the soluble fiber portions. However, this study also reported that extrusion treatment increases the total soluble fiber content by 3% [32]. The SEM-based surface analysis of both unexpanded and expanded PPW samples revealed that both the pulverization processes produced similar results respectively (Fig. 3).

Effect of Ultrafine Grinding on Particle Size Distribution

In this study, we have tried to understand and reveal the effect of ultrafine grinding treatment on the particle size distribution of the PPW by performing experiments without using an anti-caking agent or grinding aid respectively. Results obtained from the particle size analyzer were

summarized in Table 1 (where sample 1–6 and 1'–6' are processed at different time periods such as 1, 5, 8, 13, 18 and 25 min respectively). These results show that particle size of the PPW samples are negatively correlated to the grinding time under standard experimental conditions. We observed a significant difference in the particle size of the PPW particles subjected to the extrusion treatment in the initial 5 min (sample 2 and 2') of treatment (Table 1). Later, the PPW samples were subjected to different comminution treatment periods, the particle size distribution of the PPW samples (D90) ranged from sample 1 (266.31 μm) to sample 6 (42.27 μm) and sample 1' (264.62 μm) to sample 6' (45.76 μm). We have also observed that the pulverization experiments for a time of 25 min (sample 6 and 6') resulted in particle sizes of PPW ranging between 42.27 and 45.76 μm . These results indicate that the particle size was reduced by the pulverization treatment when the distribution ranges were narrowed by providing uniform fine PPW powder upon standard pulverization treatments.

PPW with varying particle sizes can be applied commercially in food industries [25, 35]. Recent studies reported on particle size fractionation of food particles, especially rice flour, for selecting various physico-chemical properties including hydration properties, rheological properties, and enzymatic starch hydrolysis [36–39]. According to Martínez et al., the extrusion treatments significantly alter the gelatinization temperatures of rice flour, which was found to be strongly dependent on the particle size of the rice flour [35].

Effect of Ultrafine Grinding on Dietary Fiber Content

In the present study, we have tried to understand the effects of different extrusion treatments on the TDF content of PPW samples. The ultrafine grinding processing method can significantly enhance the SDF composition of PPW, thus the expanded process exhibits better results than the unexpanded process. We have observed a positive correlation between the SDF composition and the degree of comminution. Results obtained from the unexpanded process showed a significant difference between sample 1 (grinded for

Table 1 Distribution of particle size D90 of processed potato peel powder

Time (min)	Unexpanded samples	Particle size (μm)	Expanded samples	Particle size (μm)
1	Samples 1	266.31 \pm 8.5 ^e	Samples 1'	264.62 \pm 8.47 ^{dD}
5	Samples 2	168.84 \pm 5.49 ^d	Samples 2'	166.37 \pm 5.15 ^{cC}
8	Samples 3	87.97 \pm 3.28 ^c	Samples 3'	86.07 \pm 3.06 ^{bB}
13	Samples 4	61.46 \pm 2.72 ^b	Samples 4'	58.96 \pm 2.69 ^{aA}
18	Samples 5	52.05 \pm 2.01 ^{ab}	Samples 5'	51.87 \pm 2.11 ^{aA}
25	Samples 6	42.27 \pm 1.62 ^a	Samples 6'	45.76 \pm 1.73 ^{aA}

Where time is represented in minutes, and the type of extrusion is unexpanded and expanded. The lower-case letters indicate 0.05 significant level, and the uppercase letters indicate 0.01 significant level respectively

1 min), sample 2 (grinded for 5 min) and sample 3 (grinded for 8 min), whereas among the extruded materials, sample 3 exhibited a significant increase of 25.9%. Conversely, the expanded process results showed no difference between sample 1' (grinded for 1 min) and sample 2' (grinded for 5 min). However, a significant difference was observed in sample 3' (grinded for 8 min) and sample 4' with an increase of 29.1%. We have also observed a highly significant difference between sample 1 (grinded for 1 min), sample 2 (grinded for 5 min) and sample 3 (grinded for 8 min), which exhibited an increase of 25.9%. A similar pattern was observed in the expanded process for sample 1' (grinded for 1 min), sample 2' (grinded for 5 min) and sample 3' (grinded for 8 min). These results suggests that TDF, IDF and SDF content of the extruded samples were unchanged, decreased and increased respectively, which might be potentially due to the melting of some insoluble arabinoxylans such as hemicellulose, pectic compounds, and partially broken water-soluble compounds. The ultrafine extrusion of samples for 8 to 13 min significantly increased the SDF ratio. We have summarized the total contents and the final SDF ratios in Table 2.

Studies have reported that processing of food materials using extrusion methods can significantly affect the dietary fiber contents of extrudates [40]. Different food processing methods such as soaking, extrusion cooking, application of temperature, and pressure strongly modifies the dietary fiber composition of the food materials, thus affecting the physico-chemical and nutritive values of the dietary fiber [41]. According to Rashid et al., extrusion treatments strongly influence the total, insoluble and soluble dietary fiber contents of wheat bran extrudates [40]. TDF can be divided into SDF and IDF based on its rate of solubility [41]. SDF mostly includes pectin, dextran, and gum. SDFs are very important in the food and pharmaceutical industries as they can improve human health by lowering blood glucose and serum cholesterol [42], whereas the insoluble dietary fiber mainly includes cellulose, part of hemicellulose, lignin, wax, and other substances. The IDF are also supplemented in the human diet to prevent gastrointestinal problems and thus improving the gut health and bowel movements [2, 42, 43].

However, studies are also being conducted to improve the SDF content and physiological function of food materials, as we know that soluble dietary fiber composition significantly affects the nutritional quality and biological activity of the food.

Effect of Ultrafine Grinding on Physico-Chemical Properties

Previous studies have reported that extrusion processes strongly modify the physico-chemical properties of maize flour [44], orange peel [43], ginseng [45], and blue corn [46]. The physico-chemical properties specifically including viscosity, chemical composition, crystal structure, thermal properties, SWC, and solubility [44]. The WHC and SWC are two main indicators used for determining the hydration characteristics of TDF. We have conducted experiments to determine the hydration characteristics of the extruded PPW samples (Tables 3, 4). These results suggest that hydration characteristics of the extruded PPW sample were positively related to the pulverization time applied during the experimentation. However, prolonged pulverization of the PPW would negatively affects the physical structure of TDF by damaging the interstitial spaces, which are potentially involved in water absorption. We have observed a significant variation in hydration characteristics of sample 1 (1 min) and sample 2 (5 min) with WHC and SWC being increased by 20.3% and 81.2% respectively.

Similar results were observed with the WHC and SWC experiments: sample 2' was significantly higher than sample 1' with 16.8% and 52.4% respectively. These results suggest that ultrafine grinding can significantly reduce the particle size of the PPW samples and increase the surface area, water binding capacity and exposure rate of the hydrophilic groups on the surface respectively. The ultrafine grinded samples were also extruded at high temperature and pressure to break the polymers, thus generating PPW material with a low degree of polymerization and high-water absorption capacities. We have observed that the hydration ability of PPW samples resulted in fine materials with good hydration,

Table 2 Soluble dietary fiber (SDF) and total dietary fiber (TDF) ratios (dry basis) of the processed potato peel ($\bar{x} \pm SE$, $n=4$)

Type	C%	Ratio%	Type	C%	Ratio%
Sample 1	3.84	9.32 ± 0.37^{aA}	Sample 1'	4.31	10.43 ± 0.41^{aA}
Sample 2	4.69	11.32 ± 0.51^{bAB}	Sample 2'	4.76	11.56 ± 0.41^{abAB}
Sample 3	4.83	11.74 ± 0.36^{bBC}	Sample 3'	5.1	12.39 ± 0.45^{bcABC}
Sample 4	5.23	12.72 ± 0.49^{bcBCD}	Sample 4'	5.59	13.46 ± 0.54^{cdBCD}
Sample 5	5.64	13.55 ± 0.59^{cdCD}	Sample 5'	6.1	14.50 ± 0.50^{deCD}
Sample 6	5.86	14.28 ± 0.60^{dD}	Sample 6'	6.37	15.50 ± 0.65^{eD}

The data is presented as a mean ($n=4$), where C% represents content %, Samples 1, 2, 3, 4, 5, 6 denote the unexpanded and Samples 1', 2', 3', 4', 5', 6' denote the expanded extrusion procedures. The lowercase letters indicate a 0.05 significance level, and the uppercase letters indicate a 0.01 significant level

Table 3 Results of ultrafine grinding (unexpanded) process on the physical and chemical properties of potato peel ($\bar{x} \pm SE$ n=4)

Project/sample	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6
WHC /%	243.12 \pm 6.69 ^{aA}	292.58 \pm 6.1 ^{bB}	302.45 \pm 8.1 ^{bcBC}	314.1 \pm 7.61 ^{bcdBC}	322.37 \pm 8.11 ^{cdBC}	328.10 \pm 8.50 ^{dC}
SWC/mg.mL ⁻¹	2.18 \pm 0.11 ^{aA}	3.95 \pm 0.19 ^{bB}	4.1 \pm 0.20 ^{bB}	4.84 \pm 0.21 ^{cBC}	5.45 \pm 0.24 ^{cC}	6.63 \pm 0.28 ^{dD}
NSAC/%	78.25 \pm 3.12 ^{aA}	91.25 \pm 3.64 ^{aAB}	99.25 \pm 4.1 ^{bcBC}	103.0 \pm 4.71 ^{bcBCD}	108.50 \pm 3.28 ^{cdCD}	118.3 \pm 3.8 ^{dD}
SAC/%	86.25 \pm 3.40 ^{aA}	116.50 \pm 4.66 ^{bB}	178.50 \pm 5.81 ^{cC}	191.0 \pm 7.70 ^{cdCD}	209.25 \pm 7.15 ^{deD}	216.75 \pm 7.80 ^{dD}
CAC pH7.0/mg.g ⁻¹	3.67 \pm 0.14 ^{aA}	5.56 \pm 0.19 ^{bB}	7.41 \pm 0.33 ^{cC}	9.70 \pm 0.28 ^{dD}	10.1 \pm 0.48 ^{deD}	10.89 \pm 0.42 ^{dD}
CAC pH2.0/mg.g ⁻¹	9.84 \pm 0.34 ^{aA}	11.11 \pm 0.40 ^{abAB}	12.21 \pm 0.50 ^{bcBC}	13.23 \pm 0.43 ^{cdCD}	14.63 \pm 0.62 ^{dD}	18.29 \pm 0.59 ^{eE}
SCAC 2.0/mg.mL ⁻¹	63.47 \pm 2.26 ^{aA}	100.84 \pm 3.29 ^{bB}	113.67 \pm 4.19 ^{cB}	132.51 \pm 3.61 ^{dC}	138.45 \pm 3.60 ^{deC}	144.92 \pm 3.71 ^{eC}
SCAC 3.0/mg.mL ⁻¹	136.34 \pm 4.22 ^{aA}	142.63 \pm 4.45 ^{abAB}	155.49 \pm 4.56 ^{bcABC}	160.54 \pm 5.1 ^{cBC}	163.1 \pm 4.66 ^{cC}	165.16 \pm 4.94 ^{cC}

The lowercase letters indicate a 0.05 significance level, and the uppercase letters indicate a 0.01 significance level

Table 4 Effect of extrusion and ultrafine grinding (expanded) on the physical and chemical properties of potato peel ($\bar{x} \pm SE$ n=4)

Project/sample	Sample 1'	Sample 2'	Sample 3'	Sample 4'	Sample 5'	Sample 6'
WHC/%	264.31 \pm 7.25 ^{aA}	308.66 \pm 8.61 ^{bB}	317.1 \pm 8.67 ^{bcB}	317.1 \pm 10.29 ^{bcB}	328.37 \pm 8.47 ^{bcB}	338.26 \pm 8.60 ^{bB}
SWC/mg.mL ⁻¹	2.88 \pm 0.13 ^{aA}	4.39 \pm 0.20 ^{bB}	5.99 \pm 0.27 ^{cC}	5.94 \pm 0.22 ^{cC}	6.1 \pm 0.27 ^{bcB}	6.83 \pm 0.27 ^{dC}
NSAC/%	88.50 \pm 3.33 ^{aA}	111.50 \pm 4.13 ^{aAB}	118.25 \pm 5.1 ^{bcB}	119.0 \pm 4.38 ^{bcB}	122.75 \pm 4.71 ^{bcB}	125.75 \pm 3.75 ^{cB}
SAC/%	112.25 \pm 4.40 ^{aA}	179.0 \pm 6.26 ^{bB}	197.25 \pm 6.29 ^{bBC}	222.5 \pm 7.15 ^{cdD}	235.25 \pm 6.34 ^{cdD}	280.0 \pm 6.72 ^{dE}
CAC pH7.0/mg.g ⁻¹	4.27 \pm 0.18 ^{aA}	6.76 \pm 0.23 ^{bB}	8.7 \pm 0.36 ^{cC}	10.88 \pm 0.51 ^{dD}	11.10 \pm 0.42 ^{dD}	11.78 \pm 0.41 ^{dD}
CAC pH2.0/mg.g ⁻¹	10.87 \pm 0.39 ^{aA}	12.31 \pm 0.50 ^{abAB}	13.66 \pm 0.60 ^{bcB}	15.58 \pm 0.63 ^{cdD}	17.53 \pm 0.63 ^{dDE}	19.2 \pm 0.77 ^{dE}
SCAC 2.0/mg.mL ⁻¹	96.80 \pm 3.34 ^{aA}	127.91 \pm 4.31 ^{bB}	137.42 \pm 3.81 ^{bcBC}	140.72 \pm 3.92 ^{bcBC}	144.39 \pm 4.56 ^{bcBC}	147.41 \pm 4.74 ^{cC}
SCAC 3.0/mg.mL ⁻¹	137.1 \pm 3.63 ^{aA}	149.34 \pm 3.94 ^{abAB}	161.13 \pm 4.58 ^{bcBC}	162.43 \pm 4.82 ^{bcBC}	165.71 \pm 4.36 ^{bcBC}	168.82 \pm 4.81 ^{cC}

The data is presented as a mean (n=4). The lowercase letters indicate a 0.05 significance level, and the uppercase letters indicate a 0.01 significance level

emulsifying, thickening and suspending capacities, which might also potentially enhance the physico-chemical properties—especially the baking properties of dough.

The absorption properties of TDF can be determined by studying the physical and chemical absorption properties. The physical absorption depends on the bulkiness of the raw material, structure, surface area, and chemical absorption depends on the dissociation of functional groups in the raw material such as hydroxyl, carboxyl, and amino groups. Results obtained in our study showed that the absorbed grease was positively correlated with the degree of comminution. We have observed a significant difference in NSAC capacity between sample 1, 2 and 3 of the unexpanded process, as sample 3 exhibited an increase of 26.8%. Similarly, with the expanded process, sample 3' exhibited a significant increase of 33.6% compared to samples 1' and 2'. The SAC results of sample 2 (unexpanded process) and sample 2' (expanded process) was found to be significantly high compared to samples 1 and 1' with 35.1% and 59.5% respectively. In short, the PPW samples extruded for 5 min exhibited extremely significant differences between NSAC and SAC properties.

Results obtained in our study also suggest that the acidity and alkalinity of the system has a great impact on the CAC

property of the extruded material. The CAC under neutral conditions (simulating the pH of the small intestine) is lower compared to acidic conditions (simulating the acidic condition of the stomach), thus varying the absorption performance. Results obtained for CAC% from the unexpanded process under neutral conditions exhibited a significant difference between samples 2 and 1 with a 51.5% increase. Under acidic conditions, there is no difference between samples 2 and 1, but sample 3 exhibited a significant increase of 24.1%. For the expanded process, under the neutral conditions, there was a significant difference in CAC properties between samples 2' and 1' with an increase of 56.2%. Under acidic conditions, there is no difference between samples 2' and 1'. However, we have observed a significant difference between samples 3' and 3 with an increase of 25.7%. The PPW samples extruded for 8 min exhibited no significant difference in CAC capacity both between the process and the system.

Earlier studies have shown that adsorbing capacities of bile acids by TDF can effectively reduce cholesterol levels in human serum, liver and can potentially prevent colon cancer [12]. Our results show that absorptivity and the concentration of sodium cholate and the time of pulverization exhibited a positive correlation with different processing

technologies, suggesting there might be a dynamic balance of the absorption capacity of bile acids by TDF. If the concentration of the system is high, its relatively high absorption capacity is contrastingly low which is mainly due to maintenance of the normal metabolism of fat in food and ensuring the normal physiological activities of the body. Tests conducted with SCAC (sodium chelate absorption capacity) at a $2.0/\text{mg mL}^{-1}$ concentration exhibited a significant difference in the unexpanded process between samples 2 and 1 with an increase of 58.9%. Similar results were obtained for the expanded process with an increase in sample 2' of 32.1%. Tests conducted with SCAC at a $3.0/\text{mg mL}^{-1}$ concentration showed a significant difference between samples 1, 2 and 4 of the unexpanded process, with the rate of increase found to be 17.7% respectively. Similarly, we have observed that samples 1' and 2' exhibited no significant difference compared to sample 3' which exhibited an increase rate of 17.6%. The PPW samples that were pulverized for 13 min, exhibited the absorption of sodium cholate by the refined material with higher significance than that of the coarse powder. We have also reported that the mean difference and

p-value comparisons between the samples 1 and 1', samples 2 and 2', samples 3 and 3', samples 4 and 4', samples 5 and 5' and samples 6 and 6' respectively (Table 5).

Comparison of Results from Processing Methods

We have observed that extrusion and ultrafine grinding techniques have a significant effect on the structure, particle size, composition, and physico-chemical properties of PPW. The results obtained in our study were consistent with the previous reports. We have extensively compared the effect expanded and unexpanded processing methods on the PPW. The particle size, proportion, hydration and adsorptive properties of the extruded materials subjected to same grinding time were observed to differ from each other. However, the extruded samples did not exhibit a significant difference ($p > 0.05$) upon comparison. The ultrafine grinding of PPW material strongly influenced the physico-chemical properties, especially the puffing effect, resulting in a significant difference in SWC, CAC (pH 7.0), SCAC ($2.0/\text{mg mL}^{-1}$). Based on the results obtained from the composition ratio,

Table 5 Comparison of the physico-chemical properties of both the expanded and unexpanded potato peel waste (PPW) samples

Project/sample	Samples 1–1'		Samples 2–2'		Samples 3–3'	
	Mean	P-value	Mean	P-value	Mean	P-value
D90	1.69	0.89	2.47	0.75	1.90	0.69
SDF ratio/%	–1.11	0.09	–0.24	0.73	–0.65	0.31
WHC/%	–21.19	0.08	–16.09	0.18	–14.59	0.26
SWC/ mg mL^{-1}	–0.70	0.00	–0.44	0.16	–1.91	0.00
NSAC/%	–0.10	0.07	–0.20	0.01	–0.19	0.03
SAC/%	–0.26	0.00	–0.63	0.00	–0.19	0.07
CAC pH7.0/ mg g^{-1}	–0.61	0.04	–1.20	0.00	–1.29	0.04
CAC pH2.0/ mg g^{-1}	–1.02	0.10	–1.20	0.11	–1.44	0.11
SCAC 2.0/ mg mL^{-1}	–33.33	0.00	–27.06	0.00	–23.75	0.00
SCAC 3.0/ mg mL^{-1}	–0.67	0.91	–6.72	0.30	–5.64	0.42
Project/sample	Samples 4–4'		Samples 5–5'		Samples 6–6'	
	Mean	P-value	Mean	P-value	Mean	P-value
D90	2.50	0.54	0.17	0.96	–3.49	0.19
SDF ratio/%	–0.74	0.35	–0.95	0.26	–1.22	0.22
WHC/%	–3.00	0.82	–6.01	0.63	–10.16	0.43
SWC/ mL g^{-1}	–1.09	0.01	–0.62	0.14	–0.20	0.62
NSAC/%	–0.16	0.04	–0.14	0.04	–0.08	0.21
SAC/%	–0.32	0.02	–0.26	0.04	–0.63	0.00
CAC pH7.0/ mg g^{-1}	–1.18	0.09	–1.01	0.17	–0.89	0.18
CAC pH2.0/ mg g^{-1}	–2.34	0.02	–2.89	0.02	–0.91	0.39
SCAC 2.0/ mg mL^{-1}	–8.21	0.18	–5.93	0.35	–2.49	0.69
SCAC 3.0/ mg mL^{-1}	–1.89	0.80	–2.69	0.69	–3.66	0.61

The data is presented as a mean ($n=4$)

D90 particle size, SDF soluble dietary fiber, WHC water holding capacity, SWC swelling capacity, NSAC unsaturated fatty acid absorption capacity, SAC saturated fatty acid absorption capacity, CAC cholesterol absorption capacity, SCAC sodium chelates absorption capacity

physico-chemical properties and SEM analysis of extruded samples, we can infer that there is no significant difference in the effect of modification between the two processes at the given time. However, this might be due to relatively lower occurrences of components like starch and other macromolecular components (which easily puff) were not observed in the PPW. Thus perhaps the relative high occurrence of TDF, protein and other substances weakened the bulking effect of the PPW. With an unexpanded comminution process for 13 min, we have observed a significant difference in the material structure, composition and its performance compared to the materials extruded using expanded process; this modification effect is in line with results from previous studies on the equilibrium with TDF composition containing 10% of SDF respectively [14].

The TDF content in the PPW was observed to be up to 40% during the production of potato granules. Application of ultrafine grinding under the influence of high temperature, pressure and shear force can reduce the material to a compact structure, with loose and scaly material. Results obtained from the SEM analysis of extruded samples showed significant changes, suggesting that proper pulverization methods can reduce the particle size of the PPW powder with a narrow distribution range, and which might result in a fine powder with good uniformity. Soluble dietary fiber is an important factor in improving the nutritional quality and biological activity of commercial food materials. The extrusion procedures reported in this study could potentially be used for increasing the ratio and content of the total SDF up to varying degrees. The PPW samples extruded and grinded for 8 to 13 min resulted in a higher proportion of SDF with better physiological functions. The hydration characteristics (WHC and SWC) of the material exhibited a positive correlation with the comminution time and the strength of the growth rate gradually reducing over time. We have also determined the hydration capacities of the extruded samples, which reported a good hydration, emulsifying, suspension and thickening properties.

The performance of the adsorbed oils showed an upward trend with the increase in degree of extrusion. The NSAC and SAC exhibited significant differences for the materials extruded for 5 min. The acidity and alkalinity of the system exhibited a great influence on the CAC% of the material; under neutral conditions the absorption capacity was lower

than the normal conditions, but with increased degrees of comminution samples exhibited higher absorption rates. The samples extruded for 8 min exhibited extremely significant CAC% properties irrespective of comminution process and system acid-base conditions. Contrastingly, SCAC content was positively correlated with the concentration of sodium cholate and the time of extrusion. The samples extruded for 13 min showed that the absorption effect of the refined material on sodium cholate was significantly higher than that of coarse powder. The PPW extruded using expanded and unexpanded procedures at the same comminution times did not exhibit significant differences in their physico-chemical properties such as particle size, SDF ratio, hydration characteristics, and absorption properties. However, the PPW samples extruded for 13 min exhibited highly significant difference from the other extruded materials, especially with the improved properties such as SEM analysis, composition ratio and physico-chemical properties (Fig. 4).

Conclusion

Studies were being conducted around the world to minimize the industrial food waste and improve the food quality. Different food processing techniques such as ultra-micropulverization, ultrahigh-pressure based treatments, freeze-pulverization, extrusion cooking, and nanotechnology-based techniques were applied industrially for enhancing the quality and physico-chemical properties of food products. Industrially, these techniques are applied either in alone or in combinations for enhancing the properties of food products. In this study, we have developed a single processing method for enhancing the physico-chemical properties of PPW. We have compared the PPW obtained from both the ultrafine grinding process and the extrusion coupled with ultrafine grinding techniques. Results obtained in our study showed that only the ultrafine grinding technique can significantly enhance the physico-chemical properties of PPW, by significantly modifying the physico-chemical properties of PPW (TDF, particle size, and SDF content). These results encourage us to conduct our future studies on applications of PPW as a food supplementing agent for enhancing the TDF and physico-chemical properties of the commercial food products.

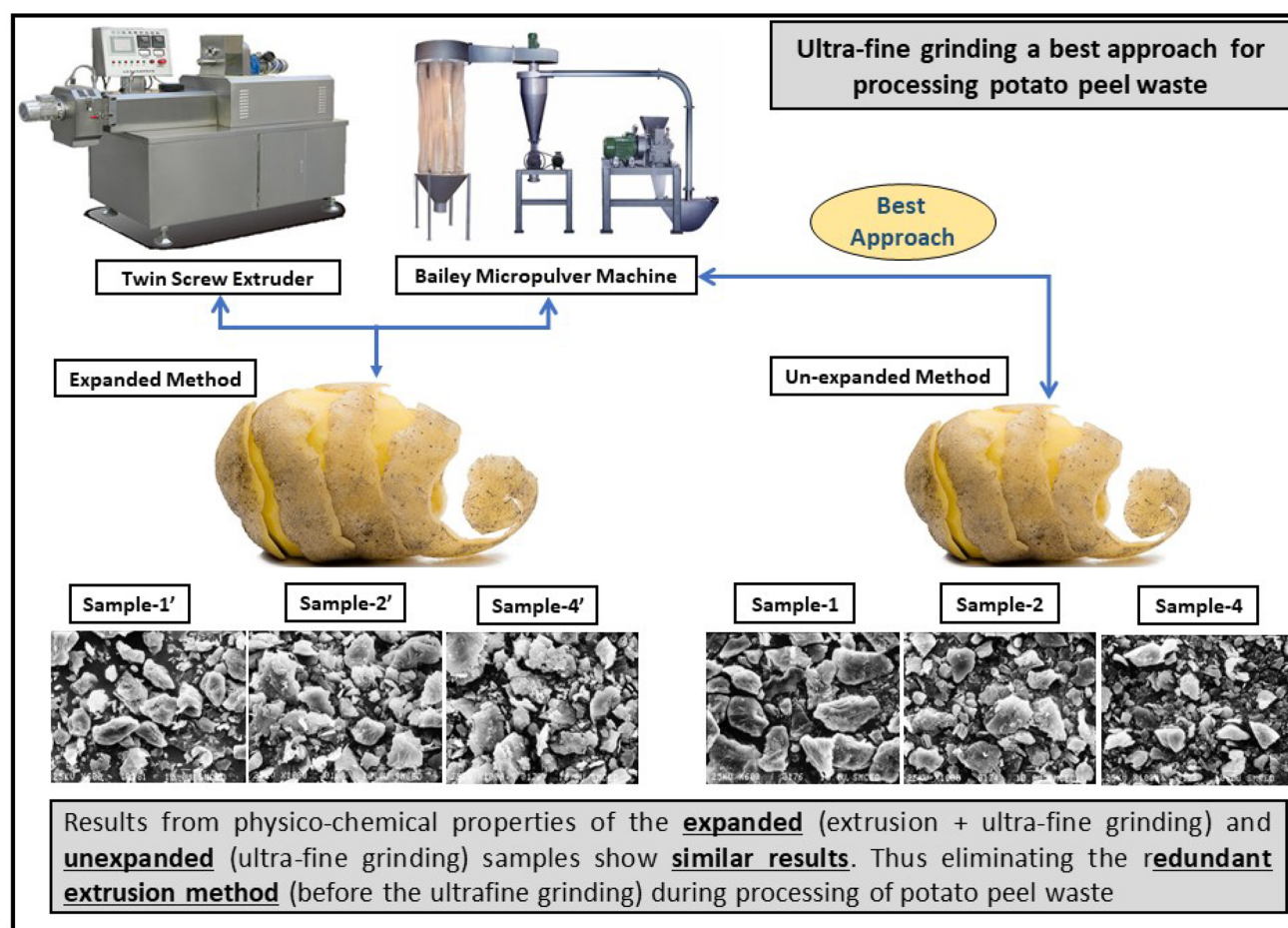


Fig. 4 Pictorial representation of comparison between the potato peel waste (PPW) extruded using expanded and unexpanded procedures

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Compliance with Ethical Standards

Conflict of interest The authors declare that they have no competing interests.

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