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Article

Chemical Composition and Functional Properties of Dietary Fiber Concentrates Obtained from Peach Palm By-Product

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The aim of this study was to produce and to characterize dietary fiber concentrates (DFCs) obtained from the peach palm by-product (PPB). Subcritical water extraction (SWE) and aqueous extractions at low pressure in magnetic (LPMS) and orbital (LPOS) stirring were carried out to DFCs production. DFCs and the untreated PPB were analyzed for composition, functional properties, scanning electron microscopy and infrared spectroscopy. The aqueous extraction treatments increased the total dietary fiber content, due to the removal of sugars (88.7-99.6%) and the partial leaching of proteins and ashes. The DFC obtained by SWE had the highest content of soluble and insoluble dietary fiber. The methods of aqueous extraction changed the structure of fiber components, that becomes more porous and fragmented, improving the functional properties, as water and oil absorption. Cellulose was the most abundant component of the samples and the alteration in its conformation was observed in the infrared spectra. The principal component analysis showed that the changes on the composition and functional properties were associated with the treatments applied. DFCs produced in this work, specially by SWE, are considered interesting alternatives to promote the utilization of peach palm by-product as a fiber-rich component.

Keywords: *Bactris gasipaes*, pressurized liquid, sugars, cellulose, principal component analysis

Introduction

The peach palm (*Bactris gasipaes* Kunth) has been cultivated in many regions of Brazil, especially for obtention of heart-of-palm, locally known as "*palmito pupunha*". This crop cultivation helps preserve native species that were used to obtain heart-of-palm. In 2018, Brazil exported approximately 290 tons of heart-of-palm, which corresponds to US$ 1.64 million in profit.[1] This product, obtained from the central part of the peach palm, is commercialized in different formats (cubes, rolls, slices) as canned or minimally processed products.[2] However, this processing generates large amounts of by-products, such as shells, sheaths and stems, that correspond, respectively, to 35, 15 and 30% of the weight of peach palm harvested.[3] These wastes are generally destinated to animal feed, however, in some cases this demand is lower than the production, causing environmental problems for the peach palm industries.

There are many studies about the valorization of by-products with focus on the extraction of active compounds, such as antioxidants,[4,5] but most studies do not consider the insoluble part, i.e., the residue of the extraction, that may contain phenolic compounds, proteins, and mainly, dietary fibers (DFs).[6-8] DFs can be used in the development of functional foods because its consumption is associated to the prevention of coronary heart diseases and some types of cancer, reduction of blood pressure and glucose levels, stimulation of beneficial intestinal bacteria growth, and they can help the weight loss due to the promotion of satiety feeling.[9]

The stem portion of heart-of-palm is an interesting source of DFs, due to its high content (62% on dry basis), however, 59% is insoluble DF, and there is 16% of soluble sugars in the by-product composition.[10] These characteristics difficult the application of this by-product, for example, in

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food formulations it is limited to the production of bakery products. In this way, processing conditions can be studied to produce dietary fiber concentrates (DFCs) from agro-industrial wastes, improving their properties. Chemical, physical and enzymatic methods can also convert insoluble fiber to soluble, which is interesting due to the beneficial effects of soluble fraction due to its fermentability and viscosity.[11,12]

In this sense, the use of subcritical water extraction (SWE) can be applied, since in addition to being considered a clean technology, it does not use organic solvents and it has high efficiency in reduced extraction time.[13] This process allows the use of temperatures above the normal water boiling point (100 °C) and bellow the critical temperature (374 °C), at pressures sufficiently capable of maintaining water in the liquid state during the process.[14] These operating conditions promote the penetration of the solvent in areas of difficult access in atmospheric conditions, facilitating the extraction of analytes retained in the pores of the matrix.[15] The application of SWE is efficient to the removal of compounds such as sugars[4,16-18] and proteins[4,14,19] of the solid matrix, being able to concentrate dietary fibers and to modify its structure, obtaining a material with better soluble/insoluble ratio.[8,20,21] However, studies that reported the characteristics of the residual material, obtained after SWE of peach palm by-product (PPB), were not found. The application of SWE or other extraction methods to obtain DFCs from PPB is an alternative to the development of new products, that tends to be economically attractive, contributing to promote the productive chains through the total use of raw material.

The aim of this study was to evaluate the effect of extraction using subcritical water, compared with aqueous extraction at low pressure using magnetic and orbital agitation, on the characteristics of dietary fiber concentrates obtained from peach palm by-product. Furthermore, physical-chemical analyses were performed to verify the effect of water extraction methods in relation to the untreated by-product.

Experimental

Materials

The by-product of heart-of-palm production (stem portion) was provided by a peach palm agroindustry (Vila Planalto, Cruzeiro do Oeste, Paraná, Brazil, latitude 23°78'13''S, longitude 53°07'63''W) and it was dried at 40 ºC for 24 h (oven with forced air circulation, Marconi, MA 035, Piracicaba, Brazil), up to reach 10 ±0.40 g of moisture, and milled (knife mill, Solab, SL-031, São Paulo, Brazil) to obtain particle size of 550 ± 70 μm. The final product was named untreated peach palm by-product (UPPB).

The reagents used in the characterization of fiber concentrates were boric acid, hydrochloric acid, sodium hydroxide, sulfuric acid, ethanol, acetone, copper sulfate, sodium carbonate, sodium phosphate and potassium sulfate (Anidrol, Diadema, Brazil); sodium bicarbonate and sodium sulfate (Nuclear, São Paulo, Brazil); protease, alpha amylase, sodium tetraborate, *m*-hydroxyphenyl and galacturonic acid (Sigma-Aldrich Chemical, Saint Louis, USA) and soybean oil (Cocamar, Maringá, Brazil).

Preparation of dietary fiber concentrates from stem portion of peach palm by-product

To obtain the dietary fiber concentrates, the UPPB was submitted to aqueous extraction, using the mass ratio of 0.05 g mL^{-1} and different extraction conditions, as defined in a previous study.[22]

The first method was carried out by subcritical water extraction using the conditions of 130 ºC, 100 bar and 90 min. To reach these parameters an experimental apparatus operated as semi-continuous mode was used, as previously reported by Iwassa *et al*.[8] Deionized water was pumped into the extraction system (1 mL min^{-1}) using a high-pressure liquid pump and this solvent was preheated, in the heating zone, before entering the extraction bed which was positioned in an oven. At the end of extraction time, the sample was cooled (10 ºC) and filtered in synthesized steel filters (Pheomenex, pore size 2 μm, diameter 1/4" and thickness 1/32").

The other assays used to obtain DFCs were carried out under lower pressure (1.013 bar) and at the same processing time used in the SWE (90 min). One of them was performed in orbital stirring (LPOS) at 150 rpm and 25 ºC using a shaker (Marconi, Ma 830/A, Piracicaba, Brazil). The last method was performed in magnetic stirring (LPMS) at 300 rpm and 100 °C using a heating plate (Ika, RCT basic, Campinas, Brazil).

The material retained in the filtration step at the end of extraction processes (SWE, LPMS and LPOS) was dried and milled as previously reported for UPPB.

Characterization analysis

The untreated peach palm by-product and the dietary fiber concentrates produced by SWE, LPMS and LPOS methods were characterized as described below.

The chemical composition analysis followed the Association of Official Analytical Chemists (AOAC)

methods: moisture by drying at 105 ºC (925.09), ashes by calcination at 550 ºC (923.03), proteins using micro Kjeldahl method and the conversion factor of 6.25 (920.87) and dietary fiber by enzymatic-gravimetric analysis (991.43).[23] Acid detergent fiber (FDA) and neutral detergent fiber (NDF) were analyzed according to the methodology of Silva and Queiroz[24] and the lignin content was obtained after hydrolysis with 72% sulfuric acid. The lignin, hemicellulose and cellulose contents were calculated from the FDA and NDF values. These results were expressed as g *per* 100 g of sample, in dry basis.

The content of uronic acids, associated to the pectin chains, were obtained by the hydrolysis of aqueous samples extract (10 mg mL^{-1}) with sulfuric acid 72% (v v^{-1}) containing sodium tetraborate ($Na_2[B_4O_5(OH)_4]\cdot 8H_2O$) 0.0125 mol L^{-1}. Then, an aliquot of 0.25 mL reacted with 0.1% *m*-hydroxyphenyl dissolved in 0.5% sodium hydroxide, and the absorbance was measured in spectrophotometer (700 Plus; Femto, São Paulo, Brazil) at 520 nm. A standard curve (coefficient of determination, $R^2 > 0.99$) was obtained with different concentrations of galacturonic acid (10 to 100 mg mL^{-1}) and the results were expressed as g of galacturonic acid content *per* 100 g of sample, in dry basis.[25]

The color analysis was carried out using the colorimeter Color Reader CR-10, Konica Minolta (Osaka, Japan), evaluating the parameters of CIE-Lab, L* (lightness), +a* (red) –a* (green), and +b* (yellow) –b* (blue). The color saturation or chroma (C*) was obtained according to equation 1,[26] and the hue angle (H) was calculated according to equation 2 when positive results were obtained for a* and b* parameters, and followed equation 3 when negative values of a* and positive values of b* were obtained.[27]

$$C^* = (a^* \times 2 + b^*2)^{1/2} \quad (1)$$

$$H\ (h / degree) = \tan^{-1}(b^*/a^*) \quad (2)$$

$$H\ (h / degree) = 180 + \tan^{-1}(b^*/a^*) \quad (3)$$

The functional properties evaluated were water solubility index (WSI), water absorption index (WAI), oil absorption index (OAI) and swelling volume (SV), according to the recommendations of Seibel and Beléia.[28] The results were expressed as g of soluble solids *per* 100 g of dried sample for WSI, g of water absorbed *per* g of dried sample for WAI, g of oil absorbed *per* g of dried sample for OAI and mL *per* g of dried sample for SV.

The samples were evaluated by scanning electron microscopy (SEM), after fixing them on a metallic support and coated with gold. A Shimadzu Quanta 250 microscope (Hillsboro, USA) was used, with an acceleration voltage of 12.5 kV for viewing and capturing images.

The spectroscopy in the infrared region with Fourier transform (FTIR), using a total attenuated reflex (ATR) sampling device, was used to evaluate the functional groups present in the samples. The spectra were obtained using a spectrophotometer (Agilent, model Cary 630, Santa Clara, USA), recorded in the spectral range of 4000 to 650 cm^{-1} in a resolution of 4 cm^{-1} and 32 scans.

Statistical analysis

The influence of the treatments and the characterization of the samples were carried out in genuine duplicate (n = 4) and the results were expressed by the mean ± standard deviation. The mean values were submitted to analysis of variance (ANOVA) at the 5% probability level, followed by the Tukey's test, using the software Statistica 7.0 (StatSoft, Inc., Tulsa, USA).[29]

The principal component analysis (PCA) was performed with the Past software (Paleontological Statistics, version 4.03).[30] The correlated dataset consisted of a matrix of 4 rows and 6 columns, considering the types of treatments and the values of chemical composition and functional properties, respectively. To define the number of extracted factors, the sets of criteria corresponding to the sum of the cumulative percentage of variance higher than 60%, eigenvalue higher than 1 and the screen test as described by the variance percentage criterion, Kaiser criterion and diagram criterion were considered of inclination, respectively.[31,32]

Results and Discussion

Chemical composition

Table 1 shows the chemical composition of the dietary fiber concentrates in comparison to the untreated by-product. After the extraction treatments, there was a reduction in the sugar content present in the UPPB of 88.72, 92.29 and 99.62% for LPOS, LPMS and SWE methods, respectively, which is an important effect, aiming its addition in food products with low energy value or for the production of supplements.

The high efficiency of the sugar removal with the application of SWE is probably due to the operational conditions applied (100 bar and 130 °C). The pressure helps in the rupture of the matrix, forcing the solvent to penetrate the solid pores and solubilizing the analytes, increasing the mass transfer of the solutes to the solvent, in addition to exercising the function of maintaining the water in liquid state, when its temperature exceeds the boiling point.[33,34] The temperature in turn, contributes to

Table 1. Chemical composition of the by-product of the peach palm before and after the aqueous extraction treatments (dry basis)

Component	SWE	LPMS	LPOS	UPPB
Total reducing sugars / (g 100 g^{-1})	0.02 ± 0.00^{d}	0.41 ± 0.01^{c}	0.60 ± 0.03^{b}	5.32 ± 0.01^{a}
Ashes / (g 100 g^{-1})	1.43 ± 0.05^{d}	2.45 ± 0.03^{c}	3.32 ± 0.11^{b}	6.40 ± 0.00^{a}
Proteins / (g 100 g^{-1})	6.50 ± 0.26^{b}	6.81 ± 0.28^{b}	6.45 ± 0.20^{b}	8.64 ± 0.02^{a}
Total dietary fiber / (g 100 g^{-1})	86.65 ± 0.35^{a}	75.15 ± 1.19^{b}	73.95 ± 1.34^{b}	54.47 ± 4.13^{c}
Insoluble dietary fiber / (g 100 g^{-1})	80.21 ± 3.00^{a}	72.40 ± 0.33^{b}	69.33 ± 0.57^{b}	52.10 ± 0.14^{c}
Soluble dietary fiber / (g 100 g^{-1})	6.20 ± 0.31^{a}	3.60 ± 0.00^{b}	3.70 ± 0.15^{b}	3.19 ± 0.55^{b}
Hemicellulose / (g 100 g^{-1})	11.25 ± 0.49^{a}	11.98 ± 0.60^{a}	10.43 ± 0.38^{a}	12.35 ± 4.60^{a}
Cellulose / (g 100 g^{-1})	52.00 ± 2.26^{a}	46.71 ± 3.52^{a}	46.00 ± 1.13^{a}	28.75 ± 2.34^{b}
Lignin / (g 100 g^{-1})	7.80 ± 0.30^{a}	6.80 ± 0.60^{a}	4.80 ± 0.00^{b}	3.39 ± 0.31^{b}
Uronic acids / (g 100 g^{-1})	2.99 ± 0.10^{a}	3.43 ± 0.04^{a}	3.27 ± 0.05^{a}	2.52 ± 0.06^{c}

Means followed by different letters (same line) indicate a significant difference ($p < 0.05$). SWE: subcritical water extraction; LPMS: low pressure magnetic stirring; LPOS: low pressure orbital stirring; UPPB: untreated peach palm by-product.

increase the diffusivity of the extraction solvent in the matrix, promoting the solubility of the analytes,[35] an effect that also favored sugar extraction in LPMS method due to the lower residual sugar content in this treatment when compared to LPOS.

Regarding the ashes content, there was a decrease of 77% (SWE), 61% (LPMS) and 48% (LPOS) in relation to the initial content of UPPB, probably due to the water solubility of mineral salts, which differs among the processing conditions.[14] Higher temperatures promote a reduction in the viscosity of the solvent, which coupled with a decrease in the dielectric constant, facilitate the diffusion of water in the matrix, promoting an increase in the solubility of these compounds in SWE and LPMS methods.[14] Yang *et al*.[21] observed a reduction of 60% in the ashes content of bamboo shoots after the treatment in subcritical water at 135 °C.

The protein (P) content was higher in the untreated by-product than in the DFCs, with a reduction of about 25% for all treatments applied, which is probably due to the solubilization of proteins in aqueous medium.[36] This process can be influenced by extrinsic factors, such as pH, ionic strength, type of solvent and process temperature,[37] and intrinsic factors, such as the proportion of hydrophilic and hydrophobic amino acids, that determine the matrix surface wettability, water adsorption, solvation and, consequently, its solubility.[38] Removal of about 60 and 70% of the protein content of soybean and red seaweed industrial solid residue by SWE was observed by Lu *et al*.[4] and Trigueros *et al*.[14] respectively, values higher than those found in this work.

There was a gradual increase in the total dietary fiber content from 38 to 59%, after aqueous extraction treatments, due to its concentration after the removal of sugars, proteins, ashes and other components. Simas *et al*.[25] reported dietary fiber content of 70.85 g 100 g^{-1} for real palm flour (leaf sheath), higher than that of UPPB (54.47 g 100 g^{-1}), however, lower than the levels obtained in DFCs (73.95 to 86.65 g 100 g^{-1}).

According to recommendations of the Food and Nutrition Board,[39] the consumption of dietary fiber for adults should be 25 g *per* day. In order to achieve this recommendation in the diet of the consumer, when formulating a food product, it would be necessary to add a higher proportion of UPPB in comparison to the DFCs. Moreover, fiber-rich products can be incorporated into food products as non-caloric agents for partial replacement of flour, fat or sugar, as enhancers of water and oil retention, in order to improve the stability of the emulsion or oxidation.[40]

All extraction methods caused an increase in the insoluble DF content, compared to UPPB, with the highest value being found in the DFC produced by SWE. SWE was the only treatment that increased the soluble DF content, whose value was approximately 94% higher than those found in the other investigated extraction methods (Table 1). According to Yang *et al*.[21] the high temperature used in the SWE (130 °C) may have hydrolysate the insoluble fraction, contributing to the increase in the soluble fraction of DF. These authors found an increase of 1.02 to 10.70 g 100 g^{-1} for soluble dietary fiber of bamboo shoots after SWE treatment at 135 °C for 30 min. Iwassa *et al*.[8] also reported that treating the asparagus by-product with subcritical water (100 °C) increased the contents of insoluble and soluble dietary fiber, with an increase more pronounced for the soluble fraction. The increase in the content of soluble DF is important due to its easer incorporation in processed foods and drinks, that is associated to its ability to forms gels and to provide viscosity.[41]

In terms of health benefits, the dietary fiber fractions complement each other in their properties. The insoluble

DF had the capacity to increase fecal volume, while soluble DF is correlated with the decrease of intestinal glucose absorption and with the reduction of cholesterol.[42] The production of fiber-rich ingredients has an increasing demand for development of functional foods with added physiological benefits.[43]

The main components of the dietary fibers, cellulose, hemicellulose and lignin, were estimated as shown in Table 1. The hemicellulose content was similar among the samples evaluated ($p > 0.05$). All aqueous extraction treatments increased the cellulose content from 60 to 81% in relation to the mean value found in UPPB. SWE and LPMS methods also increased the lignin content, by 130 and 100%, respectively when compared to UPPB. This effect may be correlated with the concentration of DFs, especially the insoluble fiber content, since cellulose and lignin are the major components of this fraction. Ciftci and Saldaña,[44] Huerta and Saldaña[45] applied subcritical water treatment in sweet blue lupine hulls and hot pressurized water in canola straw, respectively, and they reported an increase in the contents of lignin and cellulose. This effect was attributed to the non-decomposition of cellulose and lignin and their concentration after the removal of water-soluble components,[45] as also observed in the present work.

According to Bolanho *et al*.,[10] lignin was the minor component of the dietary fibers in the flours produced with by-products of peach palm, which was related to the fact that lignification occurs only in specialized cells, corroborating with the results found in this work. A study using external sheaths of peach palm treated with sodium chlorite found a higher value for hemicellulose (27.05 g 100 g^{-1}) and lower values for cellulose (44.49 g 100 g^{-1}) and lignin (3.22 g 100 g^{-1})[46] when compared to the DFC obtained by SWE.

Regarding the components of dietary fibers, cellulose was the major component in the samples evaluated, which is also known as the largest constituent of the cell wall, and its consumption can help increase the fecal volume, promoting regular bowel movements due to their insolubility in water.[47] Hemicellulose, in turn, helps to increase the number of beneficial bacteria in the intestine that bind to cholesterol, preventing its absorption.[48]

After extractions performed under different conditions, there was an increase from 19 to 36% in the content of uronic acids, when compared to UPPB. This is probably due to the hydrolysis of fiber components during extraction treatments. The content of uronic acids observed in this study, for all samples evaluated, were higher than that described by Bolanho *et al*.[10] for the flour produced with peach palm by-product (1.59 g 100 g^{-1}). The content of uronic acids is correlated to the presence of pectin, a component of the primary cell wall, linked in the cellulose and hemicellulose network.[49] Pectin, as an important component of the soluble fraction of dietary fiber, can be used in foods as an emulsifying, stabilizing and thickening agent. In addition to its technological application, it has health benefits by delaying the uptake of glucose and lipids in the bloodstream and also reducing the serum cholesterol level.[50]

Functional and color properties

The functional properties results (Table 2) showed that the values of WSI obtained for the DFCs were lower than that found in UPPB, possibly due to the removal of sugars and the partial leaching of minerals salts and soluble proteins in the aqueous extraction treatments (Table 1). A similar effect was observed by Iwassa *et al*.[8] that found higher WSI in asparagus by-product untreated than in the material processed by SWE. The thermal degradation of sugars and their high solubility in subcritical water promote changes in the functional property of the materials that undergo this treatment.[51]

The concentration of dietary fibers after extraction treatments caused an increase in the values of WAI and OAI, again with emphasis on SWE. Yang *et al*.[21] reported that treatment with subcritical water resulted in a significant improvement of these properties in bamboo shoots, which was correlated to the increase in the specific surface, enlargement of pore sizes and the exposure of more hydrophilic and hydrophobic groups of DFs. In ambient pressure processes, these alterations do not occur at the same intensity,[52] which explains the lower values of WAI and OAI found in LPMS and LPOS than those obtained for SWE treatment.

Regarding the swelling volume (SV), there was no difference ($p > 0.05$) among the extraction treatments applied, whose values were higher (25 to 35%) than the one obtained for the untreated material. The increase of this parameter was also observed by Xie *et al*.[53] that applied high pressure treatments to modify DFs from purple-fleshed potatoes, and this effect was associated to the changes in the chemical and structural nature of material.

The values observed for WAI, OAI and SV in the DFCs produced were higher than that reported by Hua *et al*.[54] and Yang *et al*.,[21] indicating that they are good DF sources and can be applied as functional food additives. WAI is an important parameter to texture stabilization and viscosity, as this parameter is related to the ability of a substance to associate with water under specific conditions; while OAI is related to the retention of aromatic compounds,[55] and it has a stabilizing effect on high-fat foods and emulsions.[56]

Table 2. Functional and color properties of the by-product of the peach palm before and after the aqueous extraction treatments

Parameter	SWE	LPMS	LPOS	UPPB
	Functional properties			
WSI / (g 100 g^{-1})	1.20 ± 0.00^{c}	3.10 ± 0.15^{b}	3.50 ± 0.15^{b}	14.90 ± 0.35^{a}
WAI / (g g^{-1})	10.52 ± 0.59^{a}	8.80 ± 0.21^{b}	7.90 ± 0.21^{c}	7.05 ± 0.10^{d}
OAI / (g g^{-1})	6.40 ± 0.10^{a}	5.60 ± 0.02^{b}	5.41 ± 0.06^{b}	3.41 ± 0.05^{c}
SV / (mL g^{-1})	13.50 ± 0.71^{a}	13.00 ± 0.71^{a}	12.50 ± 0.71^{a}	10.00 ± 0.00^{b}
	Color parameters			
L*	70.85 ± 0.25^{b}	68.89 ± 1.42^{b}	80.07 ± 0.83^{a}	80.80 ± 0.31^{a}
a*	3.12 ± 0.10^{a}	2.51 ± 0.15^{b}	1.28 ± 0.20^{d}	0.59 ± 0.11^{c}
b*	21.82 ± 0.10^{b}	23.60 ± 0.73^{a}	21.73 ± 0.61^{b}	23.90 ± 0.53^{a}
C*	31.54 ± 0.46^{a}	29.90 ± 1.41^{a}	23.40 ± 0.40^{b}	24.22 ± 0.66^{b}
H	81.90 ± 0.13^{d}	83.92 ± 0.23^{c}	93.40 ± 0.54^{a}	91.41 ± 0.25^{b}

Means followed by different letters (same line) indicate a significant difference ($p < 0.05$). SWE: subcritical water extraction; LPMS: low pressure magnetic stirring; LPOS: low pressure orbital stirring; UPPB: untreated peach palm by-product; WSI: water solubility index; WAI: water absorption index; OAI: oil absorption index; SV: swelling volume; L*: lightness; a*: red-green color; b*: yellow-blue color; C*: color saturation; H: hue angle.

In relation to the color properties (Table 2) the application of the SWE and LPMS treatments, carried out at 130 and 100 °C, respectively, caused a reduction in the luminosity (L*) compared to the other samples (UPPB and LPOS). These data indicate that the higher extraction temperatures caused darkening of the residue, probably due to the Maillard reaction, which occurs through the interaction between amino acids and reducing sugars (glucose and fructose). The values of the parameters a*, b*, C* and H changed according to the treatments applied, with a tendency to brown color and with higher color saturation after the extractions under high temperatures (SWE and LPMS); and to yellow and with less saturation in the samples untreated and obtained after LPOS treatment (performed at 25 ºC).

Color is an important attribute that can direct the application of fibers in food products. For example, the darker DFCs obtained in this work (SWE and LPMS) could be applied to products that have similar color characteristics, such as cookies, cakes, meat products, among others. And those with a lighter color (untreated and LPOS) could be applied to a greater variety of products and in higher amounts without compromising the original color.[57]

Correlation between chemical composition and functional properties

To simplify the data set related to the treatments applied to obtain the DFCs from UPPB, the PCA was performed, as can be observed in the score chart in Figure 1. For this analysis, a biplot chart with row labels were performed, considering a data set composed of a 4 × 6 matrix (rows × columns), where the rows corresponded to the treatments (LPOS, LPMS and SWE) and UPPB, and the columns referred to results of chemical composition (P, soluble dietary fiber (SDF), insoluble dietary fiber (IDF)) and functional properties (WSI, WAI and OAI).

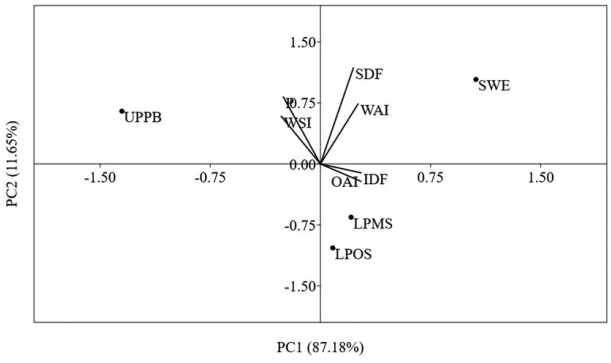

Figure 1. Principal component (PC) analysis of chemical components, proteins (P), soluble dietary fiber (SDF), insoluble dietary fiber (IDF), and functional properties, water solubility index (WSI), water absorption index (WAI) and oil absorption index (OAI) resulting from SWE, LPMS, LPOS and UPPB.

It can be seen in Figure 1 that the main components 1 (PC1) and 2 (PC2) explained 98.83% of the total variability obtained, according to the criterion of the percentage of accumulated variance. According to Kaiser's criterion, the eigenvalues considered in the PCA were higher than 1. The screen test generated an individual variance curve starting at 87.18% (PC1) with a sharp drop to 11.65% (PC2), being the most important contributors to PC1, SWE (68.29%), LPMS (13.47%) and LPOS (5.41%) and for PC2, SWE (7.17%) and UPPB (4.47%).

Positive and significant Pearson correlation was verified between the variables IDF and OAI ($r > 0.99$, $p < 0.05$). Although a high positive correlation was obtained, no

significance was found between SDF and WAI (r > 0.92, $p > 0.05$), IDF and WAI (r > 0.91, $p > 0.05$), OAI and WAI (r > 0.88, $p > 0.05$), IDF and SDF (r > 0.76, $p > 0.05$) and SDF and OAI (r > 0.73, $p > 0.05$).

Samples treated with SWE had higher values for SDF and WAI, the methods LPMS and LPOS showed similarity in relation to IDF and OAI and UPPB had the highest values for WSI and P. This effect can be evidenced by the distribution of vectors in the quadrants in the global distribution of PCA and its proximity to the points, corresponding to the applied treatments. There were negative correlations between UPPB and the parameters IDF and OAI, as well as for LPMS and LPOS and the parameters P and WSI.

In view of these results, it can be observed an increase in the values of WAI, OAI and IDF (Tables 1 and 2) as a result of the treatments applied (LPOS < LPMS < SWE), which is evidenced by the proximity of the vectors corresponding to these parameters to the treatment points. WSI, positioned in the quadrant opposite to the techniques, showed a negative correlation with the investigated treatments, with a decrease in its values on the order LPOS > LPMS > SWE. A high correlation of WSI to protein content (r > 0.98, $p = 0.01$) could be verified in the by-product, showing its dissolution after the application of different techniques, which is emphasized by the proximity of its vector to the UPPB point, that had the highest values for these parameters (Table 1).

Scanning electron microscopy

Figure 2 shows the morphological changes caused by the aqueous extraction treatments compared to the untreated

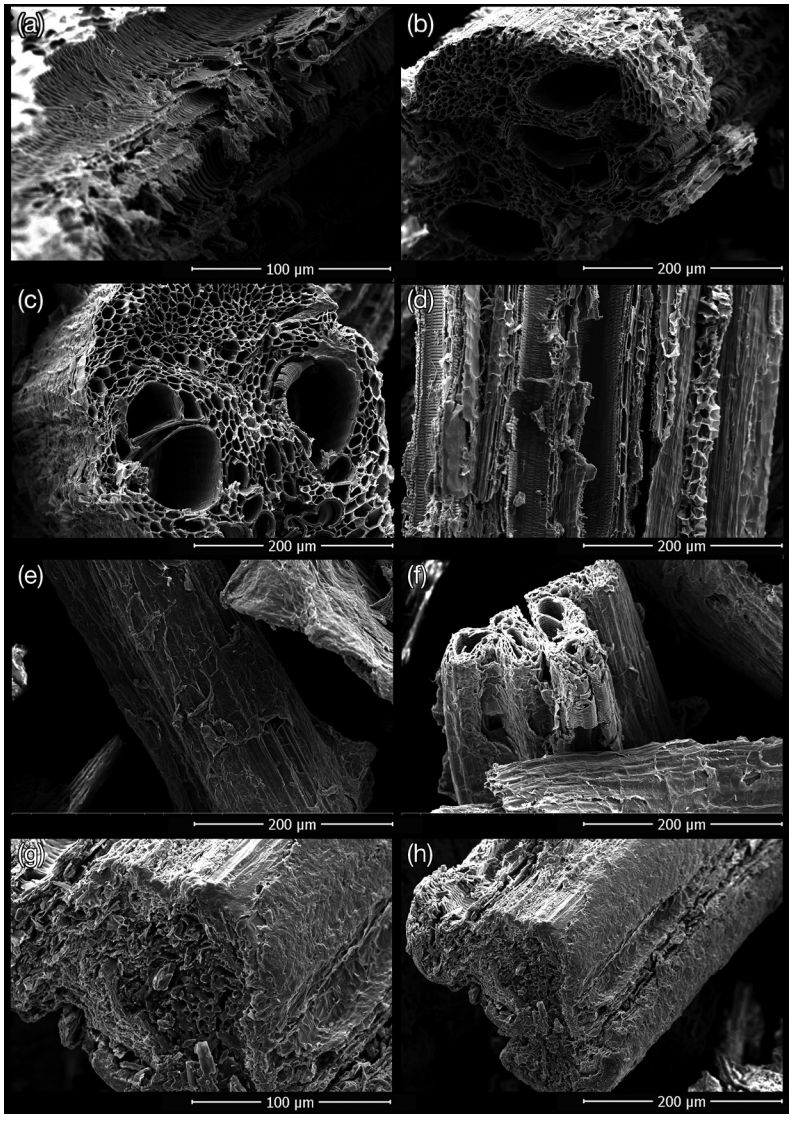

Figure 2. Electron scanning microscopy of the by-product of peach palm before and after the aqueous extraction treatments (a, b) SWE, (c, d) LPMS, (e, f) LPOS and (g, h) UPPB.

by-product. As previously reported, fibers can be seen as the main component of the peach palm by-product.

The aqueous extraction of peach palm by-product performed by SWE (Figures 2a and 2b), LPMS (Figures 2c and 2d) and LPOS method (Figures 2e and 2f) changed the surface structure of DFs, that was partially disintegrated and becomes more porous and looser when compared with UPPB (Figures 2g and 2h); this material was characterized by ordered fibers, with rigid and compact structures.

Clear cracks, larger pores in higher quantities and fragmented particles seen in the DFC obtained by SWE (Figures 2a, 2b) are associated with the degradation of cell wall polysaccharides under conditions of high temperature and pressure and the breakdown of the bundle structure of cellulose, hemicellulose and lignin. These characteristics are similar to those mentioned by Yan *et al.*[13] in a study of wheat bran based on the subcritical water method, as well as for barley and canola straw.[58] The SWE treatment increases the specific surface area of DFs, with exposition of intramolecular groups and residues, explaining the improvement of the functional properties (WAI, OAI, SV).[21]

Fourier transform infrared spectroscopy

According to Feng *et al.*,[59] the FTIR method is able to identify functional groups present in the composition of lignocellulosic fibers and Figure 3 shows the spectra obtained for each treatment applied (SWE, LPMS, LPOS) and the UPPB.

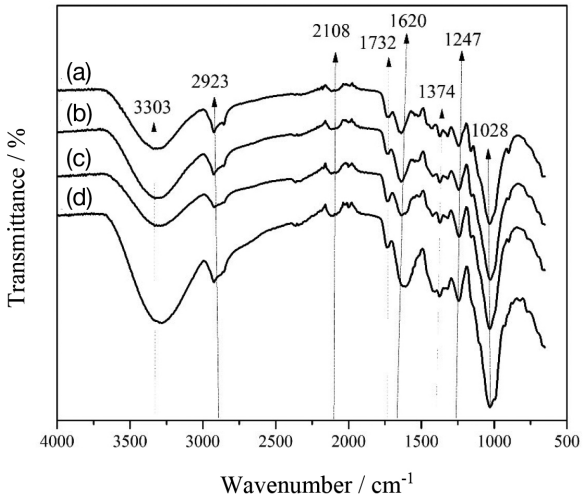

Figure 3. ATR-FTIR spectra of the by-product of peach palm before and after the aqueous extraction treatments. (a) SWE, (b) LPMS, (c) LPOS and (d) UPPB.

The broad absorption peak observed at 3000-3600 cm^{-1} in the spectra of all samples are characteristic of O–H stretching vibrations related to intermolecular hydrogen bonds of cellulose and hemicellulose.[58] The aqueous extraction methods may have caused a weaken peak intensity at 3303 cm^{-1}, especially when SWE was applied, due to the breakage of weak interaction force between hydrogen bonds in cellulose macromolecules.[21]

The peak of lower intensity at 2923 cm^{-1} may be attributed to the C–H stretching vibrations, typically of polysaccharides (cellulose and hemicellulose).[60] The peak at 1732 cm^{-1} can be a result of carbon stretching vibration of the carbonyl and acetyl groups, indicating the presence of hemicellulose.[61] The bands between 1600 to 1247 cm^{-1} correspond to bending or stretching of groups of lignin and the aromatic benzene of this molecule showed a peak at 1620 cm^{-1}.[21] The last peak at 1028 cm^{-1} may be associated to the stretching vibrations of the groups C–O and C–O–C, which is assigned to the linkage present in the cellulose, as also observed by Huerta and Saldaña[58] in a study with application of pressurized fluid treatment in barley and canola straws.

The database coupled to the ATR-FTIR system found 92% of similarity of the samples with cellulose structure, demonstrating the predominance of this component as also observed in the compositional analysis (Table 1).

Conclusions

This study contributes to know the effect of aqueous extraction methods in the composition and structure of peach palm by-product. Furthermore, the conditions applied to obtain DFCs influences its color parameters and applicability. The extraction with subcritical water is the most effective method to concentrate dietary fibers (soluble and insoluble) and to improve the functional properties. The DFCs obtained in this work are promising alternatives to enhance the use of peach palm by-product, that can be applied in the development of functional food products or used as supplements.

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