



## Effect of processing on physico-chemical characteristics of dietary fibre concentrates obtained from peach (*Prunus persica* L.) peel and pulp

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### ABSTRACT

In order to obtain rich dietary fibre concentrates with enhanced functional properties from peach peel and pulp, a treatment with ethanol (96% v/v) was performed previous to drying under 30 °C forced air convection for 7 h or under freeze drying. All the dietary fibre concentrates isolated were enriched in cell wall polymers and a high polyphenol content was associated to them. The yield of those proceeding from peel almost doubled the one of concentrates obtained from pulp and high hydration capacities were shown by all the concentrates. Fractions from peel showed the darkest colour. Oil holding capacity (1.81–2.4 g/g) was higher than the one reported in bibliography for peach bagasse (1.02 g/g) and quince wastes (1.59 g/g). Concerning the effect of the drying technique used, it was observed that air drying gave origin to pulp dietary fibre concentrates of lower oil holding capacity and less solid behaviour than freeze drying.

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### 1. Introduction

Dietary fibre (DF) exhibits a protective effect against pathologies promoted by fat and glucose (Goñi, Díaz-Rubio, Pérez-Jiménez, & Saura-Calixto, 2009; Guillon & Champ, 2000; Kay McPherson, 1982). By-products of vegetable-food processing represent a major disposal problem for the industry (Schieber, Stintzing, & Carle, 2001) and their transformation into DF concentrates may contribute to diminish the problem and to recover valuable biomass and nutrients (Gerschenson, Rojas, de Escalada Pla, & Fissore, 2009). The incorporation of these concentrates to foods can increase their DF content resulting in healthier products. They may also serve as functional ingredients to improve hydration and/or oil holding capacity, viscosity, texture, and sensory characteristics (Elleuch et al., 2011).

The area planted in Argentina with peach trees is 35,000 ha and represents the 1.4% of the world area which results in the seventh position of the ranking. The production of peaches in Argentina is of 250,000 Tn which results in the ninth position in the world production ranking. The 61% of the planted area corresponds to varieties used for

consumption as such, 38% is dedicated to varieties used for processing and the rest, to non-identified varieties. The planted area dedicated to the varieties that are consumed in fresh is mainly distributed in the provinces of Buenos Aires (10,150 ha, 46%), Mendoza (6413 ha, 29%), Córdoba (2449 ha, 11%) and Río Negro (939 ha, 4%). The national production of canned fruits has a level of 60,000 Tn and the 65% corresponds to peaches. The more important world producers of processed peaches are the USA (366,400 Tn), Greece (295,500 Tn) and Spain (187,600 Tn). According to surveys of the National Institute of Food Technology (INTA, 2000), damage due to frost produced 16.14% of the losses whereas hail is responsible for the 34.44% of the losses. Although different strategies are used to increase the yields per hectare and for diminishing the producer losses, it will be always inevitable to have an amount of fruits out of standard which cannot be sold. The industrialization of peaches also gives origin to left overs remaining from canning, juice or jam production. The transformation of left over in DF concentrates represents a strategy that can be incorporated in productive processes tending to optimize raw material use.

DF from different sources can have different metabolic and physiological effects, moreover, DF obtained from the same source can present different behaviours depending on the process applied to its obtention. Fibre properties like swelling and water retention capacity provide a general view of fibre hydration, information that is useful for fibre supplemented foods. Kinetic of water absorption provides more information on the fibre, in particular its substrate pore volume and helps the understanding of the behaviour of fibre in foods or during gut transit. The chemical composition, the anatomy and the physical characteristics of the fibres influence these values. Processes, such

*Abbreviations:* C, Air dried dietary fibre enriched products proceeding from peel; CL, Freeze dried dietary fibre enriched products proceeding from peel; P, Air dried dietary fibre enriched products proceeding from pulp; PL, Freeze dried dietary fibre enriched products proceeding from pulp; OHC, Oil holding capacity; AIR, Alcohol insoluble residue; DA, Degree of acetylation; DM, Degree of methylation; DF, dietary fibre; WSF, Water soluble fraction;  $a_w$ , Water activity; SC, Swelling capacity; WHC, Water holding capacity; WRC, Water retention capacity.

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as grinding, drying, heating or extrusion cooking for example, modify the physical properties of the fibre matrix, affecting the hydration properties (Guillon & Champ, 2000). The inclusion of DF on different food matrices can produce undesirably organoleptic changes, especially concerning colour and texture (Scott-Thomas, 2011). As a consequence, the search for alternative treatments for DF obtention from wastes as well as the diversification of sources trying to produce higher quality fibre products is still a challenge. The effect of air drying temperature on physico-chemical properties of DF of orange (Garau, Simal, Rosselló, & Femenia, 2007) and kiwifruit (Femenia et al., 2009.) was recently reported. de Escalada Pla, Uribe, Fissore, Gerschenson, and Rojas (2010) have reported the effect of the isolation procedure on the characteristics of DF concentrates obtained from quince wastes. It was observed that ethanol treatment previous to drying improved functional properties whilst water washing previous to drying produced an important loss of water soluble pectins. Grigelmo-Miguel, Gorinstein, and Martin-Belloso (1999) studied peach bagasse remaining from juice production as substrates for DF obtention and reported adequate DF contents as well as suitable functional properties for the product obtained when the bagasse was submitted to water washing and drying. Concerning peaches, it is important to remark that small changes in DF content during storage were reported by Rodríguez, Villanueva, and Tenorio (1999) whilst Muramatsu, Tanaka, Asakura, and Haji (2004) reported changes in cell wall polysaccharides during peach fruit ripening. To the best of our knowledge, no data was published regarding the effect of drying technique on physicochemical characteristics of DF obtained from peach or about possible differences on physical properties and functionality of peach DF due to the type of tissue used (peel or pulp).

The objective of the present work was to evaluate physico-chemical characteristics of DF concentrates obtained from peach peel and pulp, through the application of an ethanol treatment previous to air drying at low temperature and to compare these characteristics with those observed for products obtained through freeze drying process.

## 2. Materials and methods

### 2.1. Sample preparation

Peach (*Prunus persica* L.) variety Calred were supplied by a producer from San Pedro (Buenos Aires province, Argentina). Peaches maturity was evaluated through a firmness assay (Kader, Heintz, & Chordas, 1982) determining a value of 2 kg (over-mature peaches) and their average diameter was 7 cm. The fresh peach fruits were cleaned, peeled and the pulp was separated from the stone. Pulp and peel were submitted to a juice extraction using a domestic appliance with the purpose of eliminating great part of the water content and its solubles. Remaining solids were contacted with boiling ethanol (96% v/v) and stirred (600 RPM) for 15 min. Finally, ethanol was discarded and a portion of each remnant (pulp remnant or peel remnant) was dried under 30 °C forced air convection for 7 h obtaining the products denominated P (obtained from pulp) and C (obtained from peel). Another portion of each remnant was freeze dried (freezing under liquid nitrogen; sublimation at ambient temperature, pressure lower than 1.1 Pa for 24 h); products obtained were named PL when obtained from pulp and CL when obtained from peel.

The four fractions obtained were grounded and sifted in a mesh (sieve ASTM-USA, mesh 40). Particles with sizes smaller than 400 µm were used for the following characterization. Samples were stored at -18 °C until usage.

### 2.2. Chemicals

Deionized water (MilliQ™, USA) was used for all assays. Ethanol used was of USP grade. Chemicals were of analytical grade and, in

general, provided by MERCK Argentina (Buenos Aires, Argentina) unless stated. Gallic acid and Folin-Ciocalteu reagent were provided by Anedra S.A. (Buenos Aires, Argentina) and D-galacturonic acid, by SIGMA-Aldrich (St Louis, MO).

### 2.3. Fraction characterization

#### 2.3.1. Chemical characterization

Water content was determined in three samples of each fraction by drying at 70 °C under vacuum until constant weight.

Kjeldahl method was used for evaluating protein content (AOAC, Method 920.152, 1990).

Alcohol insoluble residue (AIR) was obtained by treating with boiling ethanol (96% v/v) each DF concentrate (de Escalada Pla, Ponce, Stortz, Gerschenson, & Rojas, 2007). Briefly, one hundred grammes of product were mixed with 350 mL of 96% v/v-ethanol solution and boiled for 15 min under stirring. The residue obtained was then extracted: (a) with 350 mL of 80% v/v-ethanol solution under boiling, for 15 min; and (b) twice with 250 mL of 80% v/v-ethanol solution under boiling, for 15 min. The insoluble residue was separated and washed with 100 mL of 80% v/v- and 100 mL of 95% v/v-ethanol solutions. Between each ethanol treatment, the suspension was filtered and the solvent was discarded. The AIR of each DF concentrate was left overnight under lab-hood to eliminate the remaining ethanol and, finally, frozen with liquid nitrogen and freeze dried. The AIR obtained was used to determine uronic acid, total (non-cellulosic) carbohydrates, cellulose and lignin contents. Hydrolysis of cellulose and non-cellulosic polysaccharides of AIR was performed according to Ng, Parr, Ingham, Rigby, and Waldron (1998) by dispersion of ≈0.3000 g of sample product into 2080 µL of 72%-sulphuric acid solution for 3 h at room temperature. The dispersion obtained for each sample was made 1 M-sulphuric acid by addition of enough deionized water in a 25.00 mL-volumetric flask and was heated at 100 °C for 2.5 h in a water-bath. After this, all dispersions were cooled, centrifuged at 12,000×g for 10 min and the supernatant was separated. The residue was washed three times with deionized water, centrifuged at 12,000×g for 10 min and finally freeze-dried and the final weight was reported as lignin.

A second procedure was carried out with other portion of ≈0.3000 g of each sample dispersed into 2080 µL of 72%-sulfuric acid solution and water was immediately added to dilute the system to a 1 M-concentration and 2.5 h of heating at 100 °C was applied. The final residue corresponded to cellulose + lignin, whilst the carbohydrate content of the supernatant (Dubois, Gilles, Hamilton, Rebers, & Smith, 1956) was constituted by non-cellulosic polysaccharides.

The third hydrolysis-procedure was performed with a new portion of each sample following the technique applied for the second procedure, but 1 h of heating at 100 °C in a water bath was applied in this case. Only the supernatant was separated for quantification as it was above indicated, and uronic acid content was determined spectrophotometrically by the method reported by Filisetti-Cozzi and Carpita (1991).

Methanol content was determined through saponification of AIR with 0.5 N-NaOH for 1 h at room temperature, followed by acidification with sulphuric acid solution needed for methanol quantification through the spectrophotometric method of Wood and Siddiqui (1971). Acetyl groups in the AIR of each DF concentrate were determined according to the method of Naumenko and Phillipov (1992). Degree of methylation (DM) and acetylation (DA) was then calculated in relation to the content of galacturonic acid for each sample (Fissore, Ponce, Stortz, Rojas, & Gerschenson, 2007).

Free sugars were determined for each DF concentrate evaluating the differences between carbohydrate content in their water soluble fraction (WSF) and carbohydrate content in the WSF of their AIR. Water-soluble fraction (WSF) was extracted by stirring the sample

( $\approx 0.5$  g) in water (50 mL) at 20 °C for 2 h as indicated by Ng and Waldron (1997).

The determination of total phenolics was carried out on each DF concentrate according to Bunzel, Ralph, Marita, and Steinhart (2000). An amount of  $\approx 0.9000$  g of sample was mixed with 50.0 mL of NaOH aqueous solution (1 mol/L) under vacuum and also protected from light, at 25 °C, for 18 h. The sample was acidified with 9.5 mL of HCl (pH < 2) and then centrifuged for 15 min at  $12,000 \times g$  (6 °C). The supernatant was used to evaluate total phenolics using the Folin-Ciocalteu technique (Singleton & Rossi, 1965). All assays were performed in duplicate.

### 2.3.2. Physical and functional characterization of fibre powders

**2.3.2.1. Water activity and colour.** Water activity ( $a_w$ ) was determined at 25 °C with a hygrometer (Aqualab, USA). Colour was measured in the CIELab space, using a Minolta device (Japan), under illuminant D65 and with the observer at an angle of 2°. Determinations were carried out three times for each fraction.

The  $\Delta E$  value was calculated as:

$$\Delta E = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}.$$

Being:

$$\Delta L^* = L^* - L_0^*$$

$$\Delta a^* = a^* - a_0^*$$

$$\Delta b^* = b^* - b_0^*.$$

Where  $L_0^*$ ,  $a_0^*$  and  $b_0^*$  are the values obtained for the freeze dried fractions and  $L^*$ ,  $a^*$  and  $b^*$  are the values of the air dried fractions.

**2.3.2.2. Specific volume, apparent density, and bulk density.** Specific volume which is defined as the inverse of apparent density ( $\rho_a^{-1}$ ) was determined by measuring the volume occupied by a weighed sample ( $\approx 5.0000$  g) using a 10.0 mL-graduated and calibrated cylinder. The bottom of the cylinder was gently tapped, on a laboratory bench, several times until there was no further decrease of the sample level (Chau Chi, Wang Yi, & Wen Yu, 2007). Determinations were carried out three times for each fraction.

For bulk density ( $\rho_b$ ) measurements, each powder was freely poured into 30-mm height and 45-mm diameter test cells avoiding vibrations that can lead to compaction. Excess was removed as described by Moreyra and Peleg (1980) and test cells filled with each powdered sample were weighed by means of an analytical balance. Determinations were carried out five times for each DF concentrate.

All assays were performed at a constant temperature of 20 °C. Mean values and standard deviation of the measurements for each fraction are reported.

**2.3.2.3. Particle size.** Particle size distribution was evaluated by laser light scattering (Mastersizer 2000E, Malvern, UK). Experiments were conducted in ethanol (96% v/v) according to Guillon and Champ (2000). Determinations were performed in triplicate for each sample (Melcion, 2000).

**2.3.2.4. Mechanical properties.** Curves obtained from compression and relaxation assays were recorded as described by de Escalada Pla et al. (2010). Briefly, DF concentrate contained in 30-mm height and 45-mm diameter test cells was compressed at a constant crosshead speed of 5 mm/min up to a force of 40 N in an Instron Universal Testing machine (Model 3345, Instron Corp., USA). The crosshead was stopped at this position and the stress was allowed to relax. The

force–deformation and the force–relaxation curves were recorded. Determinations were performed, at least, in triplicate for each sample.

Compression data were converted to density–compressive stress relationship in order to evaluate compressibility according to Eq. (1):

$$\rho = a + b \log \sigma \quad (1)$$

where  $\sigma$  is the compressive stress,  $\rho$  is the density for each deformation suffered along compression up to 40 N and  $b$  represents the compressibility of the DF powder (Moreyra & Peleg, 1980), expressing the rate with which density changed with the stress.

Relaxation data were fitted to Eq. (2):

$$\frac{F_0 \cdot t}{F_0 - F(t)} = k_1 + k_2 \cdot t \quad (2)$$

where  $F_0$  is the initial force,  $F(t)$  is the force after a time  $t$  of relaxation and  $k_1$  and  $k_2$  are fitting parameters with a constant value. The difference [ $F_0 - F(t)$ ] diminishes with  $k_2$  and  $k_1$  increase. The  $k_2$  slope was evaluated from experimental data.

### 2.3.2.5. Hydration properties

**2.3.2.5.1. Swelling capacity (SC).** An accurately weighed amount ( $\approx 0.2000$  g) of DF concentrate was placed in a graduated conical tube. Around 10.0 mL of water was added and it was hydrated for 18 h at 25 °C-constant temperature. After this time, the final volume attained by the fibre product was measured (Raghavendra, Rastogi, Raghavarao, & Tharanathan, 2004; Robertson et al., 2000). This assay was performed three times for each concentrate. Swelling capacity was calculated as:

$$SC(\text{mL/g}) = \text{Volume occupied by the sample/original sample weight.}$$

**2.3.2.5.2. Water-holding capacity (WHC).** An accurately weighed amount ( $\approx 1.0000$  g) of DF concentrate was hydrated in a graduated conical tube with 30.0 mL of water for 18 h at 25 °C. The supernatant was decanted and the sample transferred to a weighed G4-sintered glass crucible (Borosilicate glass, IVA, Buenos Aires, Argentina) allowing draining to occur. The weight of the hydrated residue was recorded. The residual dry weight was evaluated after freeze-drying.

WHC was determined three times for each concentrate and was calculated as:

$$\text{WHC}(\text{g/g}) = (\text{Hydrated residue weight} - \text{dry residue weight}) / \text{dry residue weight.}$$

**2.3.2.5.3. Water retention capacity (WRC).** Water retention capacity was determined by hydration of an accurately weighed amount ( $\approx 1.0000$  g) of each DF concentrate with 30.0 mL of water. The system was contained in a graduated conical tube and the process lasted for 18 h and was performed at a constant temperature of 25 °C. Centrifugation for 30 min at  $2000 \times g$  was then performed into the same tube. The supernatant was separated and the residue transferred to a weighed G4-sintered glass crucible (Borosilicate glass, IVA, Buenos Aires, Argentina) to drain the remaining liquid. The retained wet DF was weighed ( $R + W_2$ ) and submitted to freeze-drying. The dried residue was then weighed ( $R$ ) (de Escalada Pla et al., 2007) and WRC was calculated as:

$$\text{WRC}(\text{g water/g dried residue}) = W_2/R$$

being  $W_2$  the retained water.

The assay was performed four times for each concentrate.

**2.3.2.5.4. Kinetics of spontaneous water absorption.** Water absorption kinetics was determined in an accurately weighed amount ( $\approx 0.0200$  g) of each dried sample, by using a modified Baumann

device (Robertson et al., 2000). The spontaneous water uptake, at 25 °C, by a capillary mechanism, was evaluated through the measurement of the volume of water absorbed by the sample at different periods of time for 90 min. Kinetic assays were carried out on three samples of each fraction.

The data were fitted to the empirical equation proposed by Pilosof, Boquet, and Bartholomai (1985):

$$q_w = \frac{Q_w t}{(B_w + t)} \quad (3)$$

where  $q_w$  corresponds to the water absorbed at time  $t$ ,  $Q_w$  is the maximal water absorption capacity, also called spontaneous water binding capacity and  $B_w$  is the time needed to absorb a half of the maximal water absorption ( $Q_w/2$ ). The parameters of the equation were evaluated.

**2.3.2.6. Oil holding capacity.** Oil holding capacity (OHC) was measured according to Garau et al. (2007). Samples ( $\approx 0.2000$  g) were mixed with sunflower oil ( $\approx 1.5000$  g), left overnight at room temperature and then centrifuged ( $1500 \times g$ ; 5 min). Supernatant was decanted and sample was weighed. OHC was evaluated from the increase on weight and expressed as g of oil absorbed/g dry sample. The assay was performed in triplicate for each sample.

#### 2.4. Statistical analysis

Statistical analysis of results was performed through ANOVA (level of significance,  $\alpha$ : 0.05) followed by pairwise multiple comparisons evaluated by Tukey's significant difference test. Linear and non-linear regressions as well as statistical analyses (Sokal & Rohlf, 1980) were performed using the Statgraphics Plus package (Version 5.1, 1997–2001, Rockville, MD, USA).

### 3. Results and discussion

#### 3.1. Water activity, moisture content and physical properties

Drying process reduced  $a_w$  of the products to values below 0.6 (Table 1), rendering dried powder products with an adequate storage stability from safety view point (Adams & Moss, 1997). PL and CL moisture contents (Table 1), were significantly lower ( $p < 0.001$ ) than those obtained from fractions air dried (P and C). Possibly air drying conditions applied were not enough to remove the water remaining after ethanol treatment. Nevertheless  $a_w$  was significantly affected ( $p < 0.01$ ) only in the case of pulp DF concentrates (P and PL). C and CL concentrates presented the same  $a_w$  values but CL contained

significantly lower moisture ( $p < 0.01$ ) probably due to their different sorption behaviour.

The values corresponding to the different measurements of density can be observed in Table 1. The values of  $\rho_a$  and  $\rho_b$  for all the concentrates were significantly different ( $p < 0.05$ ) being the lowest values those from the freeze dried samples. Consequently, the specific volumes calculated were higher for PL and CL. In addition fractions proceeding from peel presented lower densities than those from the pulp. Apparent and bulk densities depend on the water content, on the chemical and physical characteristics of the solid and on the proportion of air volume which is affected by the method of drying and by the collapse of the matrix (Koç, Eren, & Ertekin, 2008). The higher density of P with respect to C can be explained, at least in part, by the higher moisture content of the former. It can be stated that P and C showed apparent densities  $\approx 20$  to 30% lower than the values reported for peach DF concentrate prepared from the drying of water washed peach bagasse by Grigelmo-Miguel et al. (1999). de Escalada Pla et al. (2010) studying DF concentrates obtained from quince wastes also observed lower values of apparent density when an ethanol treatment previous to drying was applied instead of an aqueous treatment.

Other density parameter evaluated was  $\rho_{40}$ . This was accomplished through a compression test performed till attaining a force of 40 N ( $\rho_{40N}$ ). Significant differences in this parameter were observed when comparing DF concentrates submitted to different drying techniques but not when comparing fractions obtained from pulp or peel (Table 1), being the lowest values those of freeze dried concentrates. According to Larrauri (1999) densities depend on the structural characteristics of each material, the particle size and their distribution. Table 1 also shows the particle size distribution determined through light scattering evaluating the mass median diameter ( $D_{0.5}$ ) which corresponds to the equivalent diameter for which the value of cumulative distribution is 50% (Melcion, 2000). The fact that CL had a higher  $D_{0.5}$ , means that this fraction had more particles with higher diameters than PL trend that could explain, at least in part, the lower apparent and bulk densities that CL showed when compared with PL (de Escalada Pla et al., 2010).

According to Guillon, Auffret, Robertson, Thibault, and Barry (1998) fibre structure is important in fermentation. In particular, these authors stated that to degrade polysaccharides, microbial glycosidases must have access to their substrates within the cell wall, existing a strong relationship between the total pore volume accessible to enzymes and the total pore volume accessible to bacteria. They stated that for the dietary fibres in general, the surface area available for bacteria is a major factor involved in the control of fermentation. According to this, DF concentrates with higher specific volume like CL and PL will be more prone to fermentation than C and P. And

**Table 1**

Physical properties, water content and moisture content of DF concentrates obtained from peach pulp and peel, either by drying at 30 °C for 7 h (P and C, respectively) or by freeze drying (PL and CL, respectively) in both cases after an ethanolic treatment.

	P	C	PL	CL
Moisture (g/100 g) <sup>1</sup>	11.43 ± 0.02 <sup>a</sup>	9.50 ± 0.08 <sup>b***</sup>	4.5 ± 0.1 <sup>c***</sup>	5.3 ± 0.2 <sup>d**</sup>
$a_w$ <sup>2</sup>	0.58 ± 0.04 <sup>a</sup>	0.51 ± 0.04 <sup>a</sup>	0.22 ± 0.05 <sup>b**</sup>	0.494 ± 0.001 <sup>a</sup>
Specific volume ( $\rho_b^{-1}$ ) <sup>2</sup> (cm <sup>3</sup> /g) <sup>2</sup>	2.06 ± 0.04 <sup>a</sup>	2.32 ± 0.01 <sup>a,b</sup>	2.51 ± 0.01 <sup>b</sup>	2.9 ± 0.2 <sup>c</sup>
Apparent density; $\rho_a$ (g/cm <sup>3</sup> ) <sup>2</sup>	0.486 ± 0.009 <sup>a</sup>	0.431 ± 0.002 <sup>b**</sup>	0.399 ± 0.002 <sup>c</sup>	0.35 ± 0.02 <sup>d***</sup>
Bulk density, $\rho_b$ (g/cm <sup>3</sup> ) <sup>3</sup>	0.43 ± 0.01 <sup>a</sup>	0.374 ± 0.009 <sup>b***</sup>	0.355 ± 0.002 <sup>c</sup>	0.333 ± 0.005 <sup>d</sup>
$\rho_{40N}$ (g/cm <sup>3</sup> ) <sup>4</sup>	0.47 ± 0.01 <sup>a</sup>	0.45 ± 0.02 <sup>a</sup>	0.382 ± 0.003 <sup>b***</sup>	0.39 ± 0.01 <sup>b***</sup>
$D_{0.5}$ ( $\mu$ m) <sup>5</sup>	390 ± 20 <sup>a</sup>	374 ± 1 <sup>a</sup>	301 ± 7 <sup>b**</sup>	377 ± 8 <sup>a</sup>

Different letters in the same row indicate significant differences ( $p < 0.05$ ; \*\* $p < 0.01$ ; \*\*\* $p < 0.001$ ).

<sup>1</sup> Mean and standard deviation ( $n = 2$ ) are reported.

<sup>2</sup> Mean and standard deviation ( $n = 3$ ) are reported.

<sup>3</sup> Mean and standard deviation ( $n = 5$ ) are reported.

<sup>4</sup> Density determined after compression up to a final force of 40 N. Mean and standard deviation ( $n = 5$ ) are reported.

<sup>5</sup> Mass median diameters from particle size distribution. Mean and standard deviation ( $n = 2$ ) are reported.

also fractions produced from pulp will have a higher fermentability than those coming from peel.

### 3.2. Colour

Incorporation of fibre rich products within a food system may affect the organoleptic characteristics. In fact, one of the challenges that industries faces when increasing DF content and whole grain supply in food is the change in colour and texture that these incorporations might produce (Scott-Thomas, 2011).

Table 2 shows lightness ( $L^*$ ), and the chromatic coordinates  $a^*$  and  $b^*$  for the different DF concentrates. Large significant differences were observed between pulp and peel fibre luminosity, being peel fibre fractions (C and CL) the ones with lower  $L^*$  ( $p < 0.05$ ). In addition, these fractions presented the highest ( $p < 0.001$ ) values for coordinates  $a^*$  (positive values, red colour) and  $b^*$  (positive values, yellow colour). Nevertheless, the same colour difference for P and PL and C and CL ( $\Delta E \approx 13$ ) was found allowing to conclude that the colour of fibre concentrates was influenced not only by the tissue used but also and mainly by the drying process. Since a pretreatment with boiling ethanol previous to drying was applied, it could be assumed that enzyme activity was lost and mainly, non-enzymatic browning reactions must have been responsible for colour changes (Grigelmo-Miguel et al., 1999). Garza, Ibarz, Pagán, and Giner (1999) investigated non-enzymatic browning in peach puree during heating at high temperatures (80–98 °C) and concluded that the parameter  $b^*$  was significantly reduced with heating time, especially at higher temperatures; on the contrary, parameter  $a^*$  increased during heating. Garau et al. (2007) working at temperatures between 30 and 90 °C, reported that the highest browning development was observed for pulp orange by-products dried at 30 °C, which required an extended drying period to achieve the final moisture. In this work, it was observed that air drying determined lower  $L^*$  and higher  $a^*$  and  $b^*$  than freeze drying, being all the samples in the red and yellow area of the spectrum.

### 3.3. Mechanical properties

Knowledge of the physical and mechanical properties of particulate foods is of fundamental importance for many handling processes and operations (Moreyra & Peleg, 1980). Characteristic curves obtained by compression of DF concentrates are shown in Fig. 1. The first part of the curves (0–40 N) represents the stage of compression and data for different samples was adjusted using Eq. (1). The parameter  $b$  (compressibility) obtained is reported in Table 3. Concentrates from pulp showed lower ( $p < 0.05$ ) values of compressibility than those from peel, indicating that P and PL fibres showed a lower rate of density change with the stress (Moreyra & Peleg, 1980) than C and CL, respectively, a fact that can affect DF concentrate behaviour in the digestive tract, in response, for example, to peristalsis.

The second stage of the curve shown in Fig. 1 informs the relaxation behaviour of the concentrates. The slope  $k_2$  obtained from fitting

**Table 2**

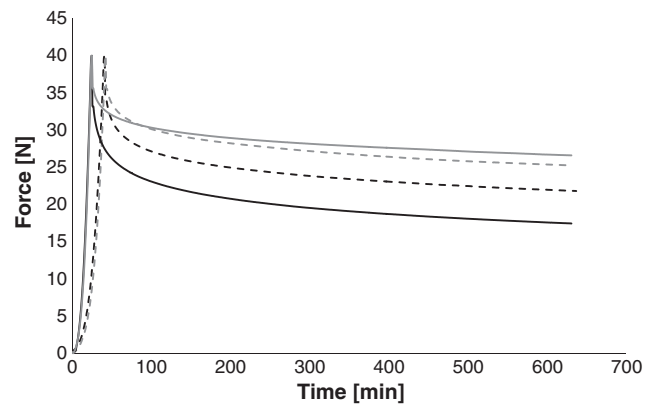
Colour parameters ( $L^*$ ,  $a^*$ ,  $b^*$ ) and colour change of DF concentrates obtained from peach pulp and peel, either by drying at 30 °C for 7 h (P and C, respectively) or by freeze drying (PL and CL, respectively) in both cases after an ethanolic treatment.

	P	C	PL	CL
$L^*$ <sup>1</sup>	76 ± 4 <sup>a</sup>	56 ± 3 <sup>b****</sup>	84 ± 2 <sup>c</sup>	69.3 ± 0.3 <sup>d</sup>
$a^*$ <sup>1</sup>	5.5 ± 0.2 <sup>a</sup>	12.2 ± 0.3 <sup>b****</sup>	2.8 ± 0.1 <sup>c****</sup>	9.8 ± 0.3 <sup>d****</sup>
$b^*$ <sup>1</sup>	29.39 ± 0.08 <sup>a</sup>	34 ± 1 <sup>b****</sup>	20.7 ± 0.5 <sup>c****</sup>	31 ± 1 <sup>a</sup>
$\Delta E^2$	12	14		

Different letters in the same row indicate significant differences ( $p < 0.05$ ; \*\*\*\* $p < 0.001$ ).

<sup>1</sup> Mean and standard deviation are reported.

<sup>2</sup> Change in colour of air dried concentrates with respect to freeze dried concentrates.



**Fig. 1.** Compression–relaxation curves of DF concentrates obtained from peach pulp (continuous line: P and PL) and from peach peel (dashed line: C and CL). In black, air dried fractions (P and C). In grey, freeze dried fractions (PL and CL).

relaxation data to Eq. (2), is reported in Table 3. This parameter must be greater than 1 ( $1 < k_2 < \infty$ ) and it can be considered as an index of how “solid” behaves the compacted specimen. A slope or  $k_2$  value equal to 1 indicates a “liquid” behaviour, that is, the stress response of the material relaxes to zero (Moreyra & Peleg, 1980). The value of  $k_2$  for the freeze dried concentrates was significantly ( $p < 0.001$ ) greater than those from air dried samples (Table 3), probably due to the lower moisture content of PL and CL (Table 1) that determined a smaller plasticization of the matrix (Moreyra & Peleg, 1980). At the same time, PL behaviour resulted slightly but significantly ( $p < 0.001$ ) “more solid” than CL. Anyhow, air dried samples showed the opposite behaviour.

### 3.4. Chemical composition

Table 4 summarizes yield and chemical composition of studied DF concentrates. The yield obtained for concentrates proceeding from peel (C and CL) almost doubled the ones of those obtained from pulp (P and PL). The four fractions were enriched in cell wall polymers, as can be observed from their alcohol insoluble residue (AIR) which comprised more than 80% (w/w) of the dried fraction. The protein ( $\approx 10\%$ ) and free sugar ( $\approx 12\%$ ) contents of different fractions did not present significant differences between fractions.

Lignin, cellulose, non-cellulosic carbohydrates as well as uronic (galacturonic) acid contents were determined on the AIR fraction of each DF concentrate. No differences were observed ( $p < 0.05$ ) when comparing AIR composition for concentrates obtained from pulp or peel. According to Kurz, Carle, and Schieber (2008), peeling decreased the acid soluble and increase the alkali/EDTA-soluble pectins of the fruits from the genus *Prunus*; in contrast they reported that other polysaccharide fractions were inconsiderably affected by peeling. In the present work, higher values were obtained for cellulose content when fractions were air dried (P and C) being P the one showing the highest content. Simultaneously, a trend to a smallest content of non-cellulosic carbohydrates was observed for air dried samples. Non significant differences were found when comparing lignin content in the concentrates. Values obtained were higher than the ones reported by Grigelmo-Miguel et al. (1999) for dietary fibre obtained from peach washed bagasse but it has to be considered that raw materials, ripening stage and type of processing affected products obtained (Femenia, Sánchez, Simal, & Rosselló, 1998a, 1998b; Grigelmo-Miguel et al., 1999; Kay McPherson, 1982).

Near 50% of the AIR of PL and CL was constituted by non-cellulosic polysaccharides (NCP) and uronic acids are the 50% of NCP (Table 4). This value was slightly but significantly reduced ( $p < 0.05$ ) in the case of C fraction, when air drying was applied. Pectins contain branched (RG I) regions, which are rich in neutral sugars (so-called “hairy

**Table 3**

Mechanical parameters calculated from compression and relaxation curves of DF concentrates obtained from peach pulp and peel, either by drying at 30 °C for 7 h (P and C, respectively) or by freeze drying (PL and CL, respectively) in both cases after an ethanolic treatment.

	P	C	PL	CL
Compressibility <sup>1</sup>	0.012 ± 0.001 <sup>a</sup>	0.022 ± 0.002 <sup>b***</sup>	0.0093 ± 0.0002 <sup>a</sup>	0.018 ± 0.002 <sup>c</sup>
$k_2^2$	1.63 ± 0.03 <sup>a</sup>	1.92 ± 0.02 <sup>b***</sup>	2.68 ± 0.06 <sup>c***</sup>	2.35 ± 0.05 <sup>d***</sup>

Mean and standard deviation are shown.

$\rho$ : g/cm<sup>3</sup>;  $\sigma$ : kgf/cm<sup>2</sup>.

Different letters in the same row indicate significant differences ( $p < 0.05$ ; \*\*\* $p < 0.001$ ).

<sup>1</sup> Slope from  $\rho = a + b \log \sigma$ ;  $\rho$ : g/cm<sup>3</sup>;  $\sigma$ : kgf/cm<sup>2</sup>.

<sup>2</sup> Slope of the normalized relaxation curve.

regions”), as well as unsubstituted HG (so-called “smooth regions”). The difference between non-cellulosic and uronic acid contents gives approximately the proportion of neutral sugars in the pectins belonging to the hairy regions of primary cell walls (Vincken et al., 2003). It can be concluded that P, C, PL and CL concentrates were enriched in cell wall polymers belonging to the primary cell wall as indicated from their high proportion of neutral sugars and that pectins found in the products isolated contained more hairy regions than those found in fibre products isolated from quince waste by de Escalada Pla et al. (2010). In addition, only slight reduction in the proportion of hairy regions occurred with the air drying process herein applied. Femenia et al. (1998a, 1998b) stated that drying causes further degradation of pectic polysaccharides and, to a minor extent, xyloglycans; more recently they also suggested that methylated pectins exhibited a higher resistance to degradation/solubilisation promoted by thermal processing (Femenia et al., 2009). Pectins from peach pulp and peel herein assayed, showed a high methylation degree (more than 90%) for all fractions and an acetylation degree ranging from 19 to 22.6%. Uronic acid and neutral sugar data are in agreement with the results published for peach by Grigelmo-Miguel et al. (1999).

Antioxidants play an important role in the prevention of oxidative stress-related diseases. Quantitatively, the main dietary antioxidants are polyphenols (PP), followed by vitamins and carotenoids (Pérez-Jiménez et al., 2008). Goñi et al. (2009) informed that PP associated with polysaccharides and proteins in cell walls are significant constituents of dietary fibre. Table 4 shows P, C, PL and CL polyphenol contents. The highest PP content was shown by CL fraction ( $p < 0.05$ ).

**Table 4**

Yield and composition of DF concentrates obtained from peach pulp and peel, either by drying at 30 °C for 7 h (P and C, respectively) or by freeze drying (PL and CL, respectively) in both cases after an ethanolic treatment.

	P	C	PL	CL
Yield <sup>1</sup>	2.75	5	2.6	4.6
Protein <sup>2</sup>	10.4 ± 0.7 <sup>a</sup>	10.5 ± 0.2 <sup>a</sup>	10.93 ± 0.07 <sup>a</sup>	11 ± 1 <sup>a</sup>
Free sugars <sup>2</sup>	13 ± 3 <sup>a</sup>	11 ± 2 <sup>a</sup>	12 ± 2 <sup>a</sup>	10 ± 1 <sup>a</sup>
Lignin <sup>2,3</sup>	12.75 ± 0.04 <sup>a</sup>	21 ± 3 <sup>a</sup>	15 ± 4 <sup>a</sup>	16.7 ± 0.3 <sup>a</sup>
Cellulose <sup>2,3</sup>	15.5 ± 0.8 <sup>a</sup>	11 ± 2 <sup>a,b</sup>	5 ± 3 <sup>b,c</sup>	2.0 ± 0.2 <sup>c</sup>
Non-cellulosic carbohydrates <sup>2,3</sup>	35 ± 6 <sup>a,b</sup>	31 ± 3 <sup>a</sup>	43 ± 1 <sup>b</sup>	45 ± 4 <sup>b</sup>
Uronic acids <sup>2,3</sup>	20 ± 3 <sup>a,b</sup>	15 ± 3 <sup>a</sup>	23 ± 3 <sup>b</sup>	20 ± 1 <sup>a,b</sup>
Degree of methylation (DM)	>90	>90	>90	>90
Degree of acetylation (DA)	19 ± 2 <sup>a</sup>	22 ± 1 <sup>a</sup>	20.6 ± 0.2 <sup>a</sup>	22.6 ± 0.9 <sup>a</sup>
AIR <sup>2,4</sup>	88 ± 5 <sup>a</sup>	83 ± 5 <sup>a</sup>	87 ± 4 <sup>a</sup>	81 ± 2 <sup>a</sup>
Total phenolic content <sup>2</sup>	0.50 ± 0.01 <sup>a</sup>	0.61 ± 0.01 <sup>a</sup>	0.6 ± 0.1 <sup>a</sup>	0.97 ± 0.05 <sup>b</sup>

Means and standard deviations are reported ( $n = 3$ ).

Different letters in the same row indicate significant differences ( $p < 0.05$ ).

<sup>1</sup> g of DF concentrates/100 g of quince pressed waste.

<sup>2</sup> g/100 g of DF concentrates.

<sup>3</sup> Components of AIR.

<sup>4</sup> Alcohol insoluble residue.

It is important to remark that even though an ethanol treatment was applied, a considerable PP content remained associated to DF concentrates P, C, PL, and CL. In addition, air drying effect on PP content was only observed on products obtained from peel.

### 3.5. Functional properties

The functional properties of DF concentrates influence their nutritional quality and their behaviour as food ingredients in product development (de Escalada Pla et al., 2007). These properties are related to the amount and characteristics of constituent polysaccharides and are influenced by porosity and particle size of the material (Femenia, Lefebvre, Thebaudin, Robertson, & Bourgeois, 1997). Drying process may alter the physicochemical properties of the products, also modifying their functional properties (Femenia et al., 2009; Garau et al., 2007).

Hydration properties of DF concentrates obtained from peach pulp and peel as well as their oil holding capacity were studied and are shown in Table 5. No significant differences were found between peel or pulp DF hydration properties but freeze drying process led to values of WHC and WRC that doubled those obtained with air drying. Swelling capacity (SC) values were also lower ( $p < 0.01$ ) when air drying process was applied to pulp (P fraction), whilst a decreasing tendency was observed for C fraction. Probably, the higher content of non-cellulosic polysaccharides of CL and PL (Table 4) jointly with

**Table 5**

Functional properties of DF concentrates obtained from peach pulp and peel, either by drying at 30 °C for 7 h (P and C, respectively) or by freeze drying (PL and CL, respectively) in both cases after an ethanolic treatment.

	Hydration properties			OHC (g/g) <sup>1,5</sup>
	SC (cm <sup>3</sup> /g) <sup>1,2</sup>	WHC (g/g) <sup>1,3</sup>	WRC (g/g) <sup>1,4</sup>	
P	29 ± 2 <sup>a</sup>	24 ± 2 <sup>a</sup>	14.3 ± 0.4 <sup>a</sup>	1.81 ± 0.02 <sup>a</sup>
C	39 ± 3 <sup>a,b</sup>	25 ± 1 <sup>a</sup>	14 ± 2 <sup>a</sup>	2.03 ± 0.03 <sup>a</sup>
PL	47 ± 4 <sup>b***</sup>	59 ± 9 <sup>b***</sup>	33 ± 2 <sup>b***</sup>	2.29 ± 0.06 <sup>b</sup>
CL	43 ± 8 <sup>b**</sup>	47 ± 2 <sup>b***</sup>	31 ± 2 <sup>b***</sup>	2.4 ± 0.1 <sup>b**</sup>
Quince waste <sup>6</sup>	11.6	15	4.8	1.59
Pumpkin peel <sup>7</sup>	22	27	26	–
Pumpkin pulp <sup>8</sup>	41.8	43	44	–
Ripe kiwi <sup>9</sup>	25	7	7	6
Orange <sup>10</sup>	20	12	12	3
Peach waste <sup>11</sup>			9.2	1.02

For comparison purposes, information for other DF products are included. <sup>6</sup>de Escalada Pla et al. (2010). <sup>7</sup>de Escalada Pla et al. (2007). <sup>8</sup>de Escalada Pla et al. (2007). <sup>9</sup>Femenia et al. (2009). <sup>10</sup>Garau et al. (2007). <sup>11</sup>Grigelmo-Miguel et al. (1999).

Different letters in the same column indicate significant differences ( $p < 0.05$ ; \*\* $p < 0.01$ ; \*\*\* $p < 0.001$ ).

<sup>1</sup> Mean and standard deviation ( $n = 3$ ) are shown.

<sup>2</sup> Swelling capacity: cm<sup>3</sup> of hydrated and swelled concentrate per gramme of dry mass.

<sup>3</sup> Water holding capacity: gramme of water per gramme of dry mass.

<sup>4</sup> Water retention capacity: gramme of water retained after centrifugation per gramme of dry mass.

<sup>5</sup> Oil holding capacity: gramme of sunflower oil retained per gramme of dry mass.

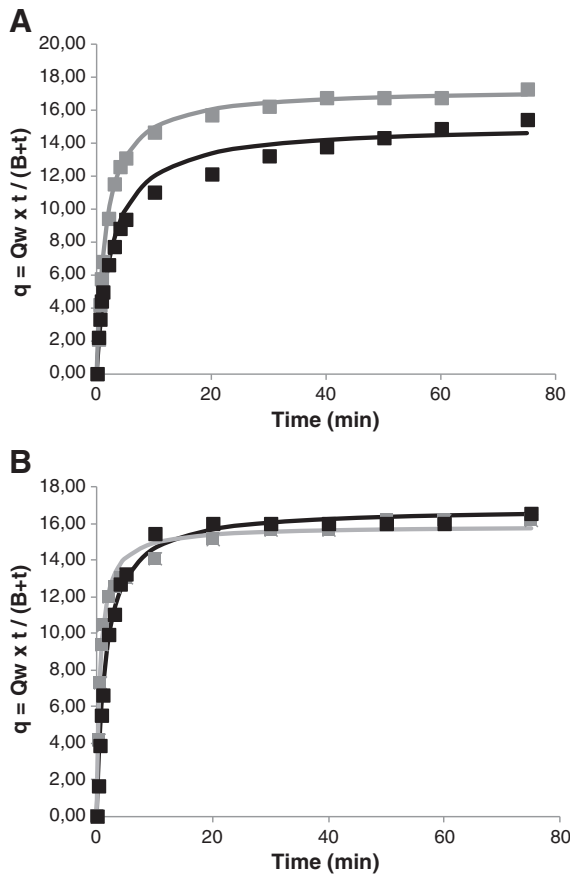


Fig. 2. Hydration kinetics for different DF concentrates. Panel A: peach peel (C and CL). Panel B: peach pulp (P and PL). In black, air dried fractions (P and C). In grey, freeze dried fractions (PL and CL). Continuous lines are the representation of the fitted model.

their higher specific volumes (Table 1) can explain the differences observed.

Water holding capacity (WHC) is defined as the quantity of water retained by the DF without the application of any external force, except gravity and atmospheric pressure (Rhagavendra et al., 2004). Thus, this parameter also includes the proportion of water loosely associated to the fibre matrix and it is therefore related to the increase in stool weight (Cadden, 1987). On the other hand, WRC is defined as the quantity of water that remains into the hydrated fibre following application of an external force like pressure or centrifugation (Rhagavendra et al., 2004). This strongly bound water has been found to have no effect on stool weight. In this sense, 51–78% of water absorbed by P and C fibre fractions (Table 5) was loosely associated to the DF matrix and this behaviour was independent of the drying process applied. Similar results were found by de Escalada Pla et al. (2010) for quince DF whilst in the case of pumpkin fibre, all water absorbed resulted to be strongly associated (de Escalada Pla et al., 2007).

Table 6  
Kinetic parameters determined from fitting data of water spontaneous absorption by DF concentrates obtained from peach pulp and peel, either by drying at 30 °C for 7 h (P and C) or by freeze drying (PL and CL) in both cases after an ethanolic treatment. Additional data was also added as reference.

	P	C	PL	CL	$Q^1$	P.Peel <sup>2</sup>	P.Pulp <sup>3</sup>
$Q_W$ (ml/g) <sup>4,5</sup>	16.9 ± 0.2 <sup>a</sup>	15.1 ± .4 <sup>b**</sup>	15.9 ± 0.21 <sup>a</sup>	17.35 ± 0.09 <sup>c**</sup>	6.6	12.5	25.5
$B_W$ (min) <sup>4,5</sup>	1.5 ± 0.07 <sup>a</sup>	2.6 ± 0.3 <sup>b***</sup>	0.62 ± 0.05 <sup>c</sup>	1.58 ± 0.04 <sup>a</sup>	0.47	13	0.1
$r^2$ (%)	99.57	97.72	98.56	99.87			

Different letters in the same row indicate significant differences ( $p < 0.05$ ; \*\* $p < 0.01$ ; \*\*\* $p < 0.001$ ).

For comparison purposes, information for other dietary fibre products are included. <sup>1</sup>de Escalada Pla et al. (2010). <sup>2</sup>de Escalada Pla et al. (2007). <sup>3</sup>de Escalada Pla et al. (2007).

<sup>4</sup>  $Q_W$ : water binding capacity;  $B_W$ : time needed to absorb  $Q_W/2$ .

<sup>5</sup> Mean and standard deviation are shown.

Oil holding capacity (OHC) was significantly higher ( $p < 0.05$ ) when freeze drying process was applied (Table 5). No differences were observed between P and C, neither between PL and CL. In general, air dehydration promoted a decrease of the OHC (Garau et al., 2007). The higher values of WHC, WRC and OHC, in general, were observed for the products PL and CL which presented the lower values of apparent, bulk density and  $\rho_{40N}$ . Higher specific volume has been associated in literature (de Escalada Pla et al., 2010; Guillon & Champ, 2000; Prakongpan, Nitithamyong, & Luangpituksa, 2002) with the ability to uptake more oil.

In Table 5 it can be observed that in spite of air drying effects, P and C concentrates presented a better functional profile than the one reported previously for DF obtained through ethanol treatment from quince wastes followed by air dehydration (de Escalada Pla et al., 2010). Similar values of SC were informed for orange peel and pulp DF by Garau et al. (2007), for kiwifruit cell wall material by Femenia et al. (2009) and for pumpkin DF rich products by de Escalada Pla et al. (2007). In addition, WRC and OHC of P and C fractions resulted higher than those reported by Grigelmo-Miguel et al. (1999) for DF obtained from peach washed bagasse.

Knowledge of kinetics for water absorption allows, for example, the determination of the time that takes fibres to sorb the quantity of water necessary for a certain process or to decide if they must be hydrated during a known time before addition to a formulation. It also helps the understanding of the behaviour of fibre in foods during gut transit. Fig. 2 shows the curves obtained by fitting to Eq. (3) the experimental data of the spontaneous water uptake during an 80 min assay. Fitting parameters and goodness of adjustment are reported in Table 6.  $Q_W$  represents the water binding capacity, whilst  $B_W$  represents the time that is demanded by DF to absorb a quantity of water equal to  $Q_W/2$ . All DF concentrates presented similar  $Q_W$  values, being the highest ( $p < 0.01$ ) the one obtained for CL fraction. As expected, freeze dried samples (PL and CL) absorbed water faster than air dried samples (P and C). For each drying treatment  $B_W$  was higher for peel. de Escalada Pla et al. (2007) observed that  $B_W$  parameter of pumpkin DF depended more on its origin than on the treatment applied, reporting that pumpkin DF from peel presented a  $B_W$  value greater in two orders with respect to that obtained from pumpkin mesocarp. They also informed that pumpkin DF from mesocarp with or without ethanol treatment presented similar values of  $B_W$ . Comparing results herein obtained with data previously reported (Table 5), allowed to conclude that the four DF concentrates isolated from peach absorbed a quantity of water that doubled the one absorbed by quince fractions isolated by de Escalada Pla et al. (2010) and absorption was faster than the one reported for DF obtained from pumpkin peel (de Escalada Pla et al., 2007).

#### 4. Conclusions

Through an ethanol treatment followed by drying, DF concentrates rich in cell wall polymers could be obtained. The fractions proceeding from peel (C and CL) presented a rendering that was the double of the one obtained from pulp (P and PL). Only a slight

reduction in pectin “hairy regions” occurred during the air drying process. A considerable polyphenol content remained associated to DF concentrates in spite of ethanol treatment. DF obtained from peel and air dried (C) showed the lowest polyphenol content.

Good hydration properties were shown by the four DF concentrates and these properties seemed to be highly influenced by drying process but not by tissue used. Oil holding capacity (OHC) decreased when air drying was used (P and C) and these concentrates presented a better functional profile than those reported previously for quince DF obtained through ethanol treatment and air drying. In addition, water retention capacity (WRC) and OHC of P and C fractions resulted higher than those reported in literature for DF obtained from peach bagasse submitted to water washing.

All the samples showed a yellow-red colour but concentrates coming from peel showed a lower luminosity and air drying produced fractions with lower luminosity than freeze drying. The relaxation responses showed that air dried DF concentrates behaved as less solid, probably due to their higher water content.

Overall, it can be concluded that ethanol treatment and a low drying temperature can be an adequate method to obtain DF concentrates with good functional properties from *P. persica* left over, and that freeze drying can produce concentrates with higher specific volume and more prone to fermentation than C and P.

As peel content might affect DF colour, it must be taken into account the peel/pulp ratio present in the peach waste processed for obtaining concentrates with adequate characteristics for their use as food ingredients.

Sorptional behaviour, phase transitions and in vitro studies of DF physiological effects are being carried on to have a more complete characterization of the products isolated.

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