Obtention of dietary fibre enriched fractions from peach bagasse using ethanol pre-treatment and microwave drying

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A R T I C L E   I N F O

A B S T R A C T

A process involving ethanol pre-treatment and subsequent microwave drying was proposed to obtain fractions enriched in dietary fibre from peach bagasse *Prunus persica* L. The processing conditions that allow to obtain samples with optimal functional properties involved the use of 4.6 mL of ethanol (96 mL/100 mL) per gram of tissue in the extraction step performed at 20 °C for 15 min and a temperature of 55.0 °C for drying in a microwave equipment working at a maximum power of 450 W. In general, high values were observed for functional properties evaluated, fact that indicates the potential of the fractions for being used for technological as well as for nutritional purposes. Physicochemical and microstructural analysis were carried out for the product obtained with the optimal process conditions, observing that the concentration of cell wall material was 80 g/100 g dry fraction and the optimum fraction also contained phenolic compounds.

1. Introduction

In Argentina, peach production for industrial use was close to 156 Gg in 2012. The waste generated from the processing and/or non-standard quality, historically has reached 5—8% of total production, according to the Ministry of Agriculture, Livestock and Fisheries of Argentina (2012). Non-standard fruits and also wastes from processing can be used for the obtention of fractions enriched in dietary fibre (DF), contributing to diminish environmental impact and improving the economic efficiency of the production chain.

Dietary fibre (DF) consists of the plant polysaccharides and lignin which are resistant to hydrolysis by digestive enzymes of man. This definition defines a macro constituent of foods which includes cellulose, hemicellulose, lignin, gums, modified celluloses,

mucilages, oligosaccharides and pectins and associated minor substances such as waxes, cutin and suberin (McCLeary et al., 2011).

The DF functional properties are related to the composition, physicochemical properties and chemical structure of the plant polysaccharides (Guillon & Champ, 2000). Processes such as drying can alter the physicochemical properties of the original products, thus modifying the functional properties of fibre fractions (Femenia, Bestard, Sanjuan, Roselló, & Mulet, 2000). De Escalada Pla et al. (2012) reported the effect of air drying and freeze drying on functional and physicochemical properties of DF obtained from peach Calred variety.

Drying is one of the most time and energy consuming processes in food industry. New techniques are permanently investigated for reducing drying time and energy consumption without affecting quality. During the past two decades, there has been an increasing interest in microwave drying because it has several advantages such as faster drying rates, shorter drying times, decreasing energy consumption and improved quality of final products (Raghavan et al., 2005). According to Murthy and Prasad (2005), the key features in the use of microwave heating is that the temperature and moisture gradients are in the same direction, being opposite to conventional heating where moisture should leave the matter against the temperature gradient. In previous works authors

realized that an ethanol treatment step prior drying enhanced functional properties of dietary fibre (De Escalada Pla, Uribe, Fissore, Gerschenson, & Rojas, 2010; De Escalada Pla et al., 2012; De Escalada Pla, Eim, Roselló, Gerschenson, & Femenia, 2015). Moreover, preliminary studies showed that the factors that most influenced the functional properties of DF when ethanol treatment was used along with microwave drying for its obtention, were ethanol/sample ratio and drying temperature (Sette, Latorre, Soria, de Escalada Pla, & Gerschenson, 2011). Microwave drying also has been contemplated in the drying up process of carrots pomace and of oranges peel and leaves according to Bejar, Kechaou, and Mihoubi (2011) and Hernández-Ortega, Kissangou, Necoechea-Mondragón, Sánchez-Pardo, and Ortiz-Moreno (2013) respectively. In the literature there is no study, to our knowledge, analysing the effect of ethanol treatment step combined with microwave drying when they are applied for the obtention from *Prunus persica* of fractions enriched in dietary fibre.

The aim of this study was to find through the response surface methodology, the ethanol/sample ratio and the microwave drying temperature that allow obtaining fractions enriched in DF with optimal yield and properties when processing involves an ethanol treatment step previous to drying. Physicochemical and also microstructural analyses were carried out on the products obtained with the optimal process conditions in order to complete their characterization.

2. Materials and methods

2.1. Materials

2.1.1. 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. All reagents used as standards for calibration curves were provided by SIGMA-Aldrich (St Louis, MO).

2.2. Methods

2.2.1. DF enriched fractions preparation
The fractions were obtained from peaches (*P. persica* L.) Jungold variety, from which stone was separated and juice was extracted. Residual peel and pulp were milled and subjected to a treatment with ethanol (96 mL/100 mL) under continuous homogenization (Sorvall Omni Mixer, USA), at 20.0 °C for 15 min. Subsequently, the mixtures were filtered and the filtrate was dehydrated using an Ethos Plus microwave equipment (Milestone, Italy) operated at a maximum power of 450 W. A drying program based on a 1 min ramp was used to reach the desired temperature and it was followed by heating at a constant temperature.

Weight loss was recorded for drying intervals of 2.5 min plus one minute of ventilation and drying was performed till the water activity of the fractions was lower than 0.6 and constant weight was attained (Muggeridge & Clay, 2001). Sample temperatures during drying step as well as ethanol/sample ratio during treatment previous to drying varied according to an experimental design (Table 1).

The fractions obtained were milled and sieved through a mesh ASTM 40 to obtain particles of sizes below 420 μm. Samples of each system, were vacuum packed in Cryovac ™ bags (Sealed Air Corporation, Argentina) and stored at −18 °C until its characterization.

2.2.2. Evaluation of fraction functional properties
Water retention capacity (WRC), retained water (RW), water holding capacity (WHC), swelling capacity (SC) as well as oil holding capacity (OHC) were determined as previously described by De Escalada Pla et al. (2010). All determinations of the properties studied were performed in triplicate.

The water soluble fraction (WSF) of DF was determined on the supernatant of the WRC determination after its freeze drying. The WSF was calculated as:

$$WSF\,(\%) = \frac{M_1}{M_2} \times 100$$

where M_1 is the mass of solids in the freeze dried supernatant and M_2 is the mass initially weighed of DF fraction on dry basis.

2.2.3. Yield and physicochemical characterization of the fractions
The determinations were performed, at least, in duplicate.

2.2.3.1. Yield evaluation.
The percent yield of DF enriched fractions was determined as the ratio between mass of DF fraction obtained after the microwave drying and mass of peach bagasse (peel and pulp) used.

2.2.3.2. Apparent density.
It was determined by measuring the volume of a weighed sample (≈ 2.0000 g) in a 5 mL graduated and calibrated tube (Chau, Wang, & Wen, 2004).

2.2.3.3. Particle size.
The size distribution of DF particles was determined using laser light scattering (Mastersizer 2000E view. 5.20, Malvern, Worcestershire, UK), according to Guillon, Auffret, Robertson, Thibault, and Barry (1998).

2.2.3.4. Moisture content and water activity.
Moisture was determined on ≈ 0.5 g samples by using infrared heating (Ohaus MB45

Table 1
Measured responses for central composite design.

Ethanol/sample ratio (mL/g)	Drying temperature (°C)	WRC (g/g)	RW (g/100 g)	WHC (g/g)	SC (mL/g)	OHC (g/g)[1]	Specific volume (mL/g)	Yield (g/100 g)	WSF (g/100 g)
5.0	70.0	19.1 ± 0.7	46 ± 2	31 ± 2	25.4 ± 0.4	1.46 ± 0.02	2.30 ± 0.01	5.3 ± 0.2	14 ± 1
5.0	50.0	23 ± 1	58 ± 2	41 ± 2	33 ± 1	1.7 ± 0.1	2.30 ± 0.04	5.1 ± 0.2	14 ± 3
3.0	70.0	21 ± 2	49 ± 2	36.7 ± 0.2	29.35 ± 0.04	1.89 ± 0.02	2.82 ± 0.01	5.9 ± 0.2	12 ± 1
3.0	50.0	17.3 ± 0.9	44 ± 3	37.4 ± 2.2	26.4 ± 0.8	1.22 ± 0.06	2.03 ± 0.01	5.2 ± 0.2	16 ± 2
4.0	60.0	23.5 ± 0.2	53.5 ± 0.5	36.1 ± 0.8	30 ± 2	1.75 ± 0.02	2.73 ± 0.01	6.2 ± 0.2	12 ± 1
4.0	40.0	19 ± 1	49 ± 2	38 ± 1	25 ± 1	1.25 ± 0.03	2.06 ± 0.02	5.0 ± 0.2	13 ± 2
4.0	80.0	22.3 ± 0.7	56 ± 1	35.1 ± 0.4	28.6 ± 0.2	1.77 ± 0.03	2.44 ± 0.04	5.3 ± 0.2	11 ± 1
2.0	60.0	20 ± 2	44 ± 2	34 ± 1	21.6 ± 0.5	1.24 ± 0.03	2.56 ± 0.01	7.3 ± 0.2	18 ± 2
6.0	60.0	21 ± 1	51.6 ± 0.2	36 ± 1	29.2 ± 1	1.32 ± 0.08	2.02 ± 0.01	5.7 ± 0.2	14 ± 2
4.0	60.0	22.2 ± 0.5	53 ± 2	34 ± 2	31.5 ± 0.6	2.05 ± 0.03	2.85 ± 0.02	6.5 ± 0.2	12 ± 2
4.0	60.0	22.1 ± 0.7	55 ± 2	35 ± 2	32.1 ± 0.7	1.98 ± 0.09	2.85 ± 0.01	5.9 ± 0.2	10.2 ± 0.6

Responses are informed as the mean and standard deviation (n = 3).
WRC: Water retention capacity, RW: Percentage of water retained, WHC: Water holding capacity, SC: Swelling capacity, OHC: Oil holding capacity, WSF: Water soluble fraction.

moisture analyzer Corporation, USA) till constant weight. The sample a_w was determined using a AQUA LAB Series 3 Quick hygrometer (Start Decagon Devices, Inc., USA).

2.2.3.5. Alcohol insoluble residue (AIR).
Alcohol insoluble residue (AIR) was obtained by treating with boiling ethanol (96 mL/100 mL) each sample according to De Escalada Pla et al. (2010).

2.2.3.6. Carbohydrate characterization, protein and lignin content.
Uronic acids, non-cellulosic carbohydrates, cellulose and lignin contents were evaluated in the AIR of the sample of interest, according to Ng, Parr, Ingham, Rigby, and Waldron (1998) by means of sulphuric acid hydrolysis. From the final residues, cellulose and lignin were determined gravimetrically, whilst the non-cellulosic carbohydrates, uronic acid as well as protein contents were determined on supernatants with the methods reported by Dubois, Gilles, Hamilton, Robers, and Smith (1956), Filisetti-Cozzi and Carpita (1991) and Lowry, Rosebrough, Farr, and Randall (1951) respectively. Methanol content as well as acetyl groups were determined in the AIR according to Wood and Siddiqui (1971) and Naumenko and Phillipov (1992) respectively. Degrees of methylation (DM) and of acetylation (DA) were then calculated in relation to the content of galacturonic acid of each sample (Fissore, Ponce, Stortz, Rojas, & Gerschenson, 2007).

2.2.3.7. Determination of phenolic compounds.
Total phenolics determination was carried out according to Bunzel, Ralph, Marita, and Steinhart (2000). Briefly 0.9000 g of fibre was mixed with 1 mol/L NaOH for 18 h under vacuum at 25 °C. Then pH was adjusted with HCl to ~2. Once centrifuged, total phenolics were determined on supernatant by Folin–Ciocalteau using galic acid as standard (Shui & Leong, 2006).

Non-ferulic phenolic compounds were determined according to Latorre, de Escalada Plá, Rojas, and Gerschenson (2013). Briefly 0.9000 g of fibre was mixed with 2 mol/L HCl and heated for 30 min in a 95 °C water-bath. After centrifugation, non ferulic phenolic contents were determined on supernatant as above indicated.

2.2.4. Microscopic characterization
Only DF samples obtained with optimal processing condition were characterized through environmental scanning microscopy (microscope model XL30-ESEM Philips, USA) at an accelerating voltage of 20.0 kV under vacuum.

2.2.5. Experimental design and statistical analysis
A central composite design (CCD) comprising two factors, with five levels for factor (−2; −1; 0; +1; +2) was used to study the effect of the ethanol/sample ratio and of the drying temperature on DF sample properties (Montgomery, 2008). Central points were performed by triplicate. Uncoded levels (Table 1) were defined based on previous work (De Escalada Pla et al., 2012; Sette et al, 2011) and resulted for ethanol/sample factors: 2; 3; 4; 5 and 6 mL/g; while for microwave drying temperature were: 40; 50; 60; 70 and 80 °C according preliminary assayed (Sette et al., 2011). The fractions were characterized through the measurement of the following properties (dependent variables): hydration properties (WRC, WHC, RW, SC), water-soluble fraction, oil holding capacity (OHC), yield and specific volume.

The experimental data were fitted to a second degree polynomial function:

$$\psi = B_0 + B_1x_1 + B_2x_2 + B_{11}x_1^2 + B_{22}x_2^2 + B_{12}x_1x_2$$

where Ψ is the dependent variable, x_1 and x_2 are the independent variables (ethanol/sample ratio and drying temperature), B_0 is the value of the response set to the design central point, B_1 and B_2 are the linear regression coefficients, B_{11} and B_{22} are the quadratic regression coefficients of each independent variable and B_{12} is the coefficient of the term of interaction between the independent variables. The analysis of variance (ANOVA) was used to evaluate the proportion of variance explained by the polynomial model obtained and was expressed through the determination coefficient (R^2). The adequacy of the model was evaluated through the P value of the test of lack of fit. The mathematical model proposed was accepted when at least one effect showed to be significant.

Response surface methodology or RSM (Montgomery, 2008) was applied, studying the fitted surface for each dependent variable which is expressed mathematically by the polynomial function obtained. The optimal conditions that maximize simultaneously the dependent variables were evaluated using the RSM and the desirability function procedure (Barros, Spacino, & Bruns, 2003). The desirability function is a useful tool that allows maximizing simultaneously several dependent variables. The basic idea of this function is to transform a multi-response problem in a problem with a unique response through mathematic transformations (Del Castillo, Montgomery, & McCarville, 1996).

Statgraphics Centurion XV (02/15/06 V, 2007) was used for the experimental design and the statistical treatment of data.

3. Results and discussion

3.1. Experimental design development

The experimental results for the dependent variables comprising the hydration properties (WRC, WHC, RW, SC), soluble

Table 2
Coefficients of second-degree polynomial function for different variables and ANOVA analysis.

Coefficients	WRC (g/g)	RW (g/100 g)	WHC (g/g)	SC (mL/g)	OHC (g/g)	Specific volume (mL/g)	Yield (g/100 g)	WSF(g/100 g)
Constant	−57.45	−95.3	−0.1694	−102.8**	−10.83	−9.22**	−3.37	70.23
LINEAR TERM								
A:Ethanol/sample ratio	16.81	39.75**	14.17	29.08**	2.72	2.08**	−0.4256*	−16.13
B: Drying temperature	1.45	2.07	0.3658**	2.41	0.2293	0.2549**	0.3490	−0.7471
SQUARED TERM								
AA	−0.5214	−1.485**	−0.0979	−1.46**	−0.1595**	−0.1259**	0.0974	1.11**
BB	−0.0048	−0.0025	0.0032	−0.0111**	−0.0010	−0.0014**	−0.0024*	0.0020
INTERACTION TERM								
AB	−0.2046**	−0.4259**	−0.2205**	−0.2652**	−0.0237*	−0.0197**	−0.0115	0.1097
EVALUATION OF ADJUSTMENT								
R^2	74.84	77.10	71.28	85.77	93.01	87.94	72.74	80.42
Lack of fit (P)	0.1849	0.0776	0.1583	0.1557	0.8417	0.1065	0.1714	0.3530

Significance levels (α): *0.1; **0.05; ***0.01.
WRC: Water retention capacity, RW: Percentage of water retained, WHC: Water holding capacity, SC: Swelling capacity, OHC: Oil holding capacity, WSF: Water soluble fraction.

fraction, oil holding capacity (OHC), yield and specific volume for each fraction obtained are shown in Tables 1 and 2 summarizes the coefficients of the second degree polynomial function proposed and the significance of each effect, the R^2 and the P corresponding to the lack of fit test.

It can be observed that all the properties evaluated showed a good fit to the proposed model (P of lack of fit was greater than 0.05) and R^2 values higher than 70% showed that the models explained more than 70% of the variability for all the variables studied. Therefore, the mathematical models adequately predicted the responses obtained with respect to variable conditions of drying temperature and ethanol/sample ratio in the range evaluated (Barros et al., 2003).

In general, it could be observed that for WRC, RW, WHC, SC, OHC and specific volume, the linear coefficients for ethanol/sample ratio and/or drying temperature, were positive (p < 0.05). In turn, the interaction effect between independent variables resulted in all cases antagonistic (p < 0.05) and quadratic effects, when significant, were negative, except for WHC, explaining in this way the shape of surfaces shown in Fig. 1, Panels a, b, d, e, f.

As can be observed in Fig. 1 (Panels a–f), at low ethanol/sample ratios the properties WRC, RW, WHC, SC, OHC and the specific volume, increased with increasing drying temperature and this behaviour reversed for high ethanol/sample ratios. In turn, at low drying temperature these properties increased with increasing

ratio of ethanol/sample and this tendency reversed at higher drying temperatures.

On the other hand, yield and WSF showed different trends. The yield decreased with the increase of the ethanol/sample ratio for all temperature levels and showed a negative linear coefficient for this factor (Table 2); it showed a negative and significant quadratic coefficient for drying temperature which explains the curvature observed and the maximum for intermediate temperatures (Fig. 1g). The WSF increased with decreasing temperature at low ethanol/sample ratios and the trend was reversed at high ethanol/sample ratios (Fig. 1h). Otherwise at low temperatures there was an increase in the soluble fraction with decreasing ethanol/sample ratios and the trend was reversed at higher temperatures; the square effect of ethanol sample ratio was positive and significant (Table 2) fact that can explain the curvature observed in the response surface.

The direct treatment of vegetable tissues with hot ethanol produces the isolation of the cell wall material (Renard, 2005). Higher ethanol/sample ratios produced lower yield. But, if simultaneously, lower drying temperatures were applied, WRC, RW, WHC, SC, OHC and specific volume showed higher values. Similar trends were observed by the authors with DF obtained from other tissues (De Escalada Pla, Ponce, Stortz, Gerschenson, & Rojas, 2007, 2010). On the other hand, at higher ethanol/sample ratio, when drying temperature increased, those properties presented a clear
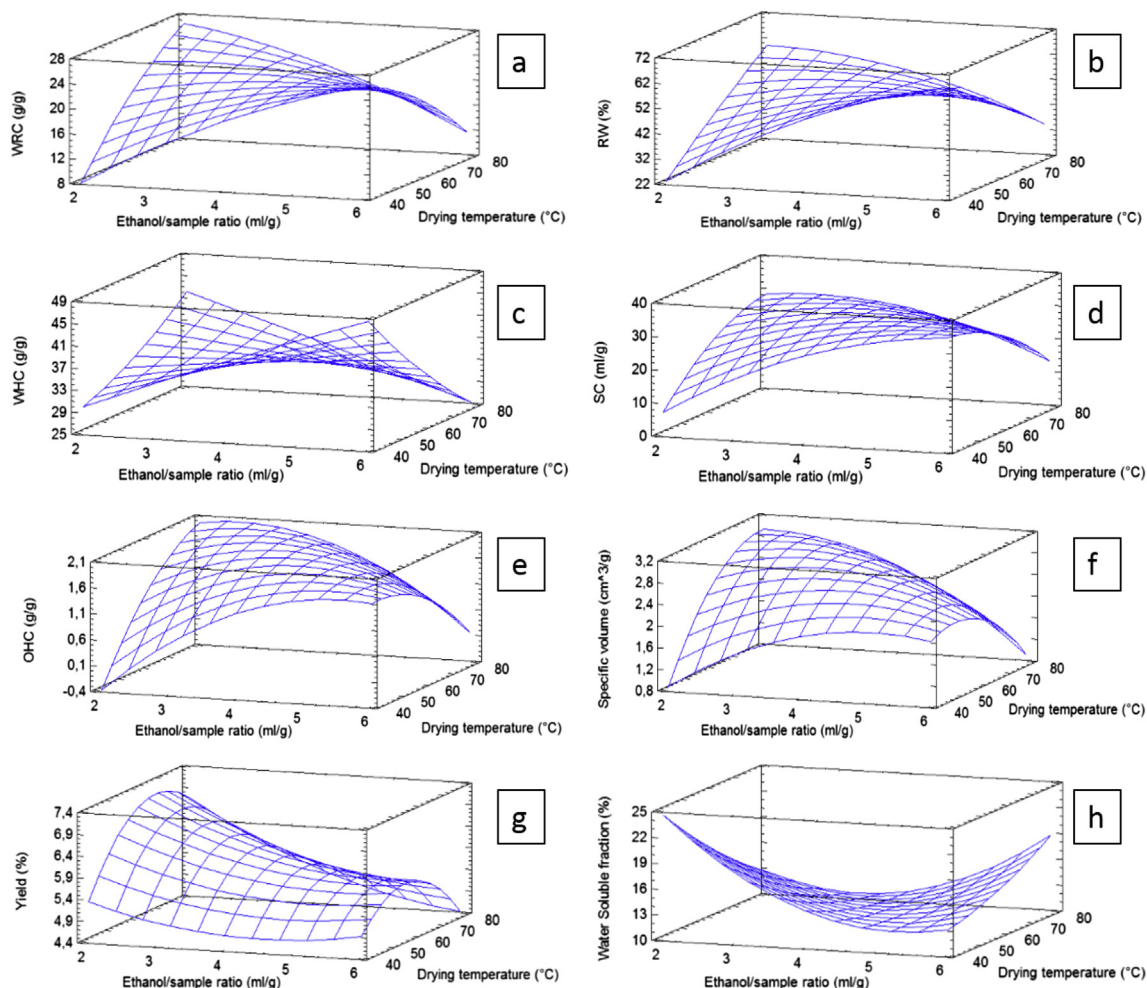

Fig. 1. Response surfaces representing the effect of the ethanol/sample ratio and the microwave drying temperature on: (a) Water retention capacity, WRC; (b) Percent water retained, RW; (c) Water holding capacity, WHC; (d) Swelling capacity, SC; (e) Oil holding capacity, OHC; (f) Specific volume; (g) Yield; (h) Water soluble fraction, WSF.

decrease and, simultaneously, WSF increased (Fig. 1). Probably, the thermal degradation of DF polysaccharides could account for this trend (Kivelä, 2011) in the range of drying temperatures herein assayed.

When low ethanol/sample ratios were applied, free glucose or soluble compounds of low molecular weight could still remain in the fractions isolated determining higher yield and higher WSF but, in general, it was observed a worst functional behaviour. This can be ascribed to matrix collapse and tissue shrinkage that can occurred in the presence of low molecular carbohydrates (Gerschenson, Bartholomai, & Chirife, 1981; Guillon et al., 1998) or to the activity of cell wall hydrolytic enzymes (Van Koningsveld et al., 2002) that catalysed the degradation of polysaccharides (Hamalainen & Reinikainen, 2007) when low drying temperatures were used. On the other hand, when higher drying temperatures were used, enzyme denaturation might have occurred and, consequently, a better functional profile can be observed (Fig. 1, Panels a–f).

3.2. Optimization

3.2.1. Optimization of each property studied

Hydration properties, oil holding capacity, particle size and specific volume were studied because of their importance for the physiological and/or technological functionality of the fractions (Guillon & Champ, 2000). Table 3 summarizes the factor (ethanol/sample ratio and drying temperature) levels that optimized the different functional properties studied and it can be observed that, in general, hydration properties showed an optimum value for an ethanol sample ratio of 6 and a drying temperature of 40 °C. The criteria for optimization was to maximize the responses due to the importance they have for the functionality of the DF fractions.

3.2.2. Multiple response optimization

The desirability function procedure was used to find the factor levels that allow to maximize simultaneously different properties. The analysis was performed in three different ways. At first, the eight properties (Group 1: WRC, RW, WHC, SC, OHC, specific volume, yield and soluble fraction) were analysed simultaneously. Then, two groups of properties were considered: one of them (Group 2) was formed by the hydration properties WRC, RW, WHC and SC and the other group (Group 3) comprised the properties OHC, yield, specific volume and water-soluble fraction. The properties were grouped in this way with the idea of evaluating the conditions for maximising all the properties together or to privilege certain group of properties that were more important for a certain technological application.

When all properties were analysed together (Group 1), optimal conditions statistically recommended were an ethanol/sample

ratio of 2 mL/g and a drying temperature of 78.2 °C and the desirability function (D) reached a value of 0.83. Similar values were observed for the optimisation of the properties of Group 3 (2 mL/g; 70.9 °C; D: 0.82). However, properties comprised in Group 2 would reach optimal values using an ethanol/sample ratio of 5.3 mL/g and a drying temperature of 45 °C with D: 1.00.

In order to experimentally verify the results obtained, two additional experiments were carried out using conditions statistically recommended for properties of Group 1-Group 3 (2 mL/g; 78.2 °C) and for Group 2 (5.3 mL/g; 45 °C). The experimental results concerning the evaluation of the properties for these two new fractions are shown in Table 4.

These two additional systems were incorporated to the first experimental design with eleven systems, obtaining a new design comprising thirteen systems. For the new experimental design, the coefficients of the polynomial, the R^2 coefficient and the lack of fit test for each property were evaluated and are reported in Table 5. As can be observed, the P values for the lack of fit test were greater than 0.05 for all the responses. But the model was not adequate for the water soluble fraction because it could not explain at least 70% of the variability. As a consequence, the optimization was performed maximizing seven of the eight properties initially proposed and optimal conditions involved an ethanol/sample ratio of 4.6 mL/g and a drying temperature of 55.0 °C and the desirability function showed a value of 0.75. The drying curve for the optimal process is shown on Fig. 2. With these conditions, 53 min of total drying time were needed to achieve desired diminution of water activity.

The fraction rich in peach DF obtained using these last conditions was characterized and the results are shown in Table 6 along with the values for the properties that had been statistically predicted. It is important to remark that values reported in this table, in general, are not as high as those estimated by RSM when properties were optimized individually (Table 3), but with the exception of RW, they are similar to or higher than those reported in Table 4 for the properties of group 1 and group 2. Moreover, these results are among the highest values reported in Table 1. Values obtained for WRC are higher than those reported for fractions enriched in dietary fibre obtained from Calred peaches by De Escalada Pla et al. (2012) when an ethanol treatment and air drying was used for isolation. The WHC has a similar value to the one reported by De Escalada Pla et al. (2012) for fractions air dehydrated or freeze-dried but it is higher than the one reported by Grigelmo et al. (1999) for a peach dietary fibre (DF) concentrate obtained from

Table 3
Optimization of responses estimated by the response surface methodology.

Functional property	Ethanol/sample ratio (mL/g)	Drying temperature (°C)	Optimum value
WRC (g/g)	2.0	80.0	26.68
RW (g/100 g)	6.0	40.0	66.16
WHC (g/g)	6.0	40.0	48.20
SC (mL/g)	6.0	40.0	34.19
OHC (g/g)	2.6	80.0	2.04
Specific volume (mL/g)	2.1	78.2	2.96
Yield (g/100 g)	2.0	67.2	7.12
WSF (g/100 g)	2.0	40.0	24.46

WRC: Water retention capacity, RW: Percentage of water retained, WHC: Water holding capacity, SC: Swelling capacity, OHC: Oil holding capacity, WSF: Water soluble fraction.

Table 4
Comparison of results obtained experimentally, with those estimated by multiple response surface analysis for properties of groups one and two.

Property	Group 1		Group 2	
	Experimental results	Estimated by multiple response surface analysis	Experimental results	Estimated by multiple response surface analysis
WRC (g/g)	22 ± 3	26.16	24.4 ± 0.2	23.69
RW (g/100 g)	55 ± 4	58.00	57.3 ± 0.8	59.94
WHC (g/g)	35 ± 2	41.58	41 ± 2	42.55
SC (mL/g)	28 ± 2	28.70	27.6 ± 0.4	33.08
OHC (g/g)	1.50 ± 0.07	1.95	1.22 ± 0.01	–
Specific Volume (mL/g)	2.30 ± 0.01	2.96	2.12 ± 0.01	–
Yield (g/100 g)	6.0 ± 0.2	6.83	4.5 ± 0.1	–
WSF (g/100 g)	10 ± 3	13.21	17 ± 1	–

Mean and standard deviation are reported (n = 3).
WRC: Water retention capacity, RW: Percentage of water retained, WHC: Water holding capacity, SC: Swelling capacity, OHC: Oil holding capacity, WSF: Water soluble fraction.

Table 5
Coefficients of second-degree polynomial function for different variables and ANOVA analysis. Experimental design with thirteen systems.

Coefficients	WRC (g/g)	RW (g/100 g)	WHC (g/g)	SC (mL/g)	OHC (g/g)	Specific volume (mL/g)	Yield (g/100 g)	WSF (g/100 g)
Constant	−52.79	−85.5	11.90	−90.81	−9.092	−7.907	−0.5875	64.87
LINEAR TERM								
A:Ethanol/sample ratio	15.75*	36.63**	10.84	24.76**	2.166**	1.706**	−1.297**	−13.73
B: Drying temperature	1.381*	1.946	0.2047**	2.278	0.2081*	0.2375**	0.3146**	−0.7002**
SQUARED TERM								
AA	−0.6491*	−1.571**	−0.3079	−1.46**	−0.1742	−0.1475***	0.0703	1.012*
BB	−0.0057*	−0.0035	0.0014	−0.0118**	−0.0012*	−0.0015***	−0.0027	0.0018
INTERACTION TERM								
AB	−0.1658**	−0.364***	−0.1329**	−0.2**	−0.0128	−0.0102**	0.0064	0.0910
EVALUATION OF ADJUSTMENT								
R^2	72.67	80.21	73.33	73.37	85.3	83.09	76.34	64.47
Lack of fit (P)	0.2116	0.1197	0.1816	0.1358	0.5808	0.1128	0.234	0.2266

Significance levels (α): *0.1; **0.05; ***0.01.
WRC: Water retention capacity, RW: Percentage of water retained, WHC: Water holding capacity, SC: Swelling capacity, OHC: Oil holding capacity, WSF: Water soluble fraction.
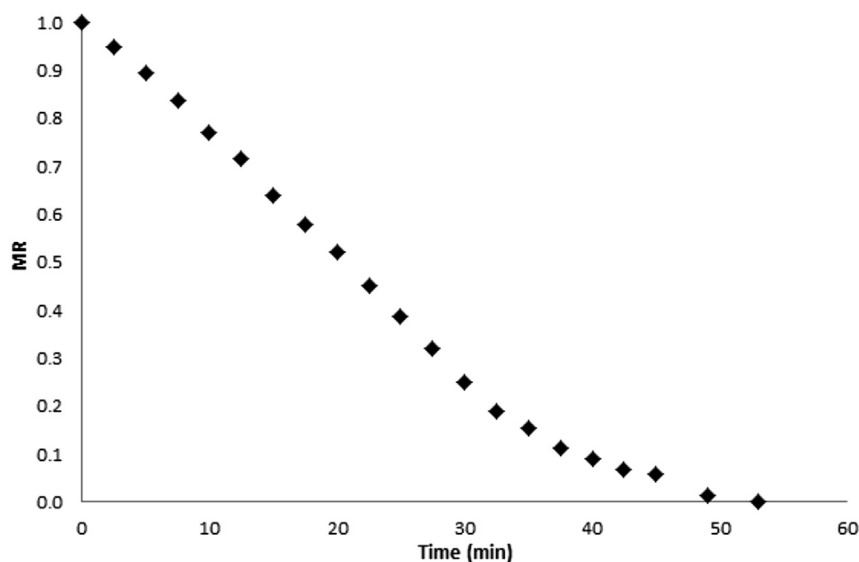
Fig. 2. Drying curve for the optimum process conditions (ethanol/sample ratio of 4.6 mL/g and a drying temperature of 55.0 °C). MR (Moisture ratio) = $(m-m_f)/(m_o-m_f)$; m: moisture at time t, m_f: final moisture, m_o: initial moisture.

peach wastes (12 g/g). Additionally, SC exceeded the value reported by Robertson et al. (2000) as a typical value for a DF concentrate (20 mL/g). The specific volume had a value of 2.81 mL/g for DF fraction isolated with the optimal conditions and was in the range of the one reported by De Escalada Pla et al. (2012) for fibres from Calred peaches and freeze drying treatment (2.5–2.9 mL/g). The OHC values (1.93 g/g) are comparable to the value of 1.8 g/g reported by De Escalada Pla et al. (2010) for DF fractions isolated from quince waste and higher than those found by Gómez, Jiménez, and Rupérez (2010) for DF isolated from edible seaweed in north-eastern spanish coast (from 1.32 to 1.61 g/g).

3.3. Additional characterization of peach DF obtained with optimal conditions

For a more complete characterisation of the fraction obtained under optimal conditions, chemical characterisation, particle size evaluation and microscopic observation were performed.

3.3.1. Particle size distribution
Particle size distribution, measured after the final grinding and sieving processes, showed values of D [0.5] of 258 (±9) μm and a Sauter diameter of 134 (±1) μm. The particle size may have an

important role, controlling a number of events occurring in the digestive tract such as the transit time, fermentation, and faecal excretion. It is important to note that the particle size may vary during the transit in the digestive tract as a result of mastication, grinding in the stomach and bacterial degradation along bowel (Guillon & Champ, 2000).

Table 6
Comparison of experimental results obtained using an ethanol/sample ratio of 4.6 mL/g and a drying temperature of 55 °C, with those estimated by multiple response surface analysis.

Property	Experimental results	Estimated by multiple response surface analysis
WRC (g/g)	22.6 ± 0.8	22.69
RW (g/100 g)	54 ± 2	54.43
WHC (g/g)	35 ± 1	37.35
SC (mL/g)	31 ± 1	31.23
OHC (g/g)	1.9 ± 0.2	1.77
Specific volume (mL/g)	2.81 ± 0.07	2.58
Yield (g/100 g)	6.2 ± 0.3	5.60

Mean and standard deviation are reported (n = 3).
WRC: Water retention capacity, RW: Percentage of water retained, WHC: Water holding capacity, SC: Swelling capacity, OHC: Oil holding capacity.
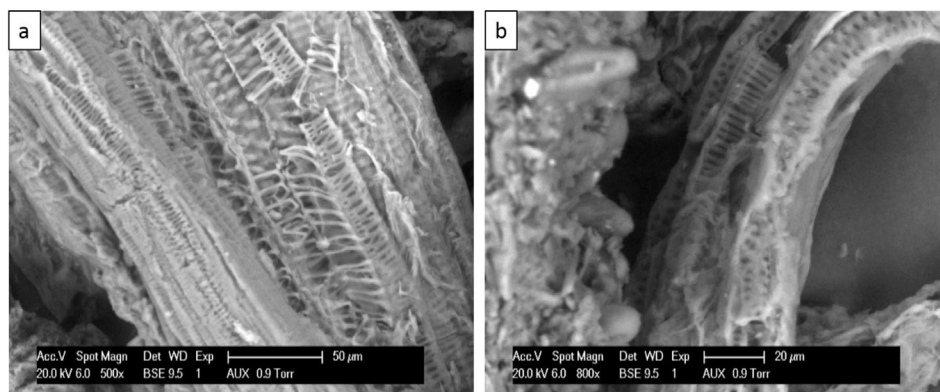
Fig. 3. Scanning electronic microscopy of DF fractions obtained from *Prunus persica* L. a: magnification 500×; b: magnification 800×.

3.3.2. Microscopic observation

Microscopic observation of DF with two different magnifications of 500× and 800× was performed. In all fields and at both magnifications, it could be observed, the presence of vascular tissue areas that remained after the ethanol and drying treatments, indicating the preservation, in part, of the histological structure (Fig. 3). Jarvis (2011) stated that the nutritional value of cell walls depends on the extent to which the walls remain physically intact during the digestion process. As a consequence, the integrity observed allows to conclude that the treatment was mild helping to obtain fractions with potential physiological value.

3.3.3. Chemical characterisation

The fraction obtained from peach presented a content of 80 g of AIR per 100 g fraction (dry basis), denoting that the sample was constituted mainly by cell wall polymers. The moisture content was 5.03 g/100 g (wet basis). The water activity (a_w) was 0.366 (±0.004), value that assures safety storage of the product at room temperature. Fraction enriched in cell wall polymers was constituted by 11 g/100 g (dry basis) of lignin, 7.0 g/100 g (dry basis) of protein, 11 g/100 g (dry basis) of cellulose. The non-cellulosic carbohydrates (HC) were present in a high proportion, 65 g/100 g fraction (dry basis), fact that reveals its hydrophilic chemical nature, which is coherent with the high value observed for hydration properties. Uronic acid content was 12 g/100 g fraction (dry basis) and pectins presented a high methylation degree (>50%) and a degree of acetylation of 15.7%. Grigelmo et al. (1999) reported values between 5.7 and 7.4 g of lignin per 100 g of fibre concentrates obtained from peach bagasse. Pagán, Ibarz, Llorca, Pagán, and Barbosa-Cánovas (2001) reported a content of 7.5 g/100 g for protein and 18.4 g/100 g for uronic acid in the dry residue obtained after peach juice extraction.

The total phenolic content was 710 mg/100 g of dry fraction, 66% of which corresponded to non ferulic phenolics. The total phenolic content was significantly higher than the content found in 100 mL of orange juice, or red wine, which are in the order of 62.56 and 195 mg/100 mL, respectively, according to Bravo, Goya, and Lecumberry (2007). De Escalada Pla et al. (2012) found a total phenolic content of 500 and 610 mg/100 g of dry fibre in samples of pulp and peel peach drying with air convection respectively. The content of phenolic compounds is important in relation to the antioxidant activity associated with them. Hayat et al. (2010) indicated that appropriate microwave treatment could be an efficient process to liberate and activate the bounded phenolic compounds and to enhance the antioxidant activity of citrus products.

3.4. Conclusions

A process involving ethanol pre-treatment and subsequent microwave drying was studied to obtain fractions enriched in DF from *P. persica* variety Jungold. Processing conditions for the obtention of fractions with optimal yield and functionality could be determined using the response surface methodology and the desirability function.

The optimal conditions found involved the use of 4.6 mL of ethanol (96 mL/10 mL) per gram of tissue in the treatment step at 20.0 °C for 15 min, and a drying temperature of 55.0 °C in the microwave equipment. These conditions allowed obtaining yields of 6.2 g of fraction per 100 g of tissue; the fraction was constituted mainly by cell wall material and the major components were non-cellulosic carbohydrates. It was also observed an interesting content of phenolic compounds, which might provide antioxidant properties to this fraction. High values of hydration properties and oil holding capacity were observed.

An ethanol pre-treatment followed by microwave drying showed to be an adequate process for obtaining fractions enriched in cell wall polymers with good functional properties. Chemical composition, conservation of tissue structure, low particle size and high specific volume probably determined the high values of the hydration properties and oil holding capacity, contributing to its technological and physiological potential.

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