### **Open Access**

Paula Sette, Jhon Edinson Nieto Calvache, Marcelo Soria, Marina de Escalada Pla, Lía N. Gerschenson\*

# Impact of different factors on the yield and properties of fractions enriched in dietary fiber isolated from peach (*Prunus persica* L.) residues

DOI: 10.1515/opag-2016-0006 received January 18, 2016; accepted June 21, 2016

Abstract: The industrialization of peaches gives origin to leftover remaining from fruit canning, juice or jam production. The transformation of leftover to dietary fiber (DF) concentrates represents a strategy that can be incorporated in productive processes tending to optimize raw material use. In the present research, DF was obtained from peach (Prunus persica L.) using an ethanol treatment followed by dehydration. The influence of four factors: time and temperature of ethanol treatment prior to drying step, ratio of ethanol to sample and drying temperature using microwave or convective drying on different properties (yield, hydration properties, oil holding capacity and apparent density) of the concentrate obtained, were studied through a two level orthogonal design. Yield ranged between 4.38-7.27 g/100g for all the conditions explored. Time and temperature of the ethanol extraction affected properties when a subsequent convective drying was employed. The effect of the ethanol/sample ratio and of the temperature of drving on oil holding capacity and hydration properties was mainly detected on samples dried by microwave technique. The results obtained

provide insight into the effect of processing of plant residues on the properties of DF concentrates obtained and on their potential performance as ingredients or additives for the food industry. It could be concluded that the drying technique is a key factor in relation to the properties of dietary fiber enriched fractions isolated from peach. Microwave drying allowed to produce fractions with functional properties that can be modulated through the use of different relations of ethanol to sample ratio and drying temperatures.

**Keywords:** Dietary fiber enriched fractions, convective drying, microwaves, functional properties

### **1** Introduction

According to Spinner (2013), the manufacture process leads to unused ingredients, unfinished products, trimming, peels and other food wastes while different sectors have common barriers to divert food wastes that include transportation, liability and lack of recycling opportunities.

On the other hand, the emission of carbon dioxide in the Mauna Loa Observatory reached a record value of 400 ppm in the last years (NOAA Research, 2013). The landfills collaborate to this trend. As a consequence, it can be concluded that it is important to manage food waste for having it out of landfills while adding value and applying it to food purposes.

Dietary fiber (DF) exhibits a protective effect against pathologies promoted by fat and glucose (Goñi et al., 2009). According to current recommendations (Food and Nutrition Board, 2005), the average daily requirement of DF is 25 g per day for women younger than 50, 21 g per day for women older than 50; 38 g per day for men younger than 50, and 30 g per day for men older than 50.

<sup>\*</sup>Corresponding author: Lía N. Gerschenson, Departamento de Industrias, FCEN, UBA, Ciudad Universitaria, (1428) Ciudad Autónoma de Buenos Aires, Argentina, Tel.: 54 11 4576-3366 / 3397, Fax.: 54 11 4576-3366, E-mail: lia@di.fcen.uba.ar

Member of the National Research Council of Argentina (CONICET) **Paula Sette, Jhon Edinson Nieto Calvache, Marina de Escalada Pla,** Industry Department, School of Natural and Exact Sciences (FCEN), Buenos Aires University (UBA)

Marina de Escalada Pla, Member of the National Research Council of Argentina (CONICET)

Jhon Edinson Nieto Calvache, Fellow of the National Research Council of Argentina (CONICET)

Marcelo Soria, Instituto de Investigaciones en Biociencias Agrícolas y Ambientales – INBA (CONICET), School of Agronomy (UBA)

<sup>(</sup>cc) BY-NC-ND © 2016 Paula Sette *et al.* published by De Gruyter Open

This work is licensed under the Creative Commons Attribution-NonCommercial-NoDerivs 3.0 License.

DE GRUYTER OPEN

The industrialization of peaches gives origin to leftover remaining from fruit canning, juice or jam production. The transformation of leftover to DF concentrates represents a strategy that can be incorporated in productive processes tending to optimize raw material use.

DF obtained from the same source can present different behaviors depending on the process applied to its obtaining (de Escalada Pla et al., 2012). DF properties like swelling and water retention capacity, specific volume, chemical composition, can be affected by processes such as grinding, drying, heating or extrusion cooking (Guillon and Champ, 2000) conditioning its nutritional properties and technological applications.

Ethanol treatment as an stage for DF production had been previously proposed with the purpose of enzyme inactivation as well as elimination of low molecular weight compounds (de Escalada Pla et al., 2010 and 2012). Microwave drying have achieved considerable attention in the recent past, gaining popularity because of its advantages over conventional heating such as reducing the drying time of biological material with small quality loss (Arslan and Özcan, 2010). Maskan (2001), had reported that microwave application improves product qualities, such as aroma and results in faster and better rehydration compared with hot air drying process.

The aim of the present work was to evaluate the effect of different factors on the yield and properties of DF enriched fractions obtained from peach (*Prunus persica* L.) residues, through an ethanol treatment followed by convective air or microwave drying. A two level orthogonal design with four central points was performed in two blocks in order to evaluate the effect of four factors, time and temperature of ethanol treatment, ratio of ethanol to sample and drying temperature, on the properties of DF with the purpose of providing insight into the effect of processing of plant residues on the performance of biobased products obtained as ingredients or additives for the food industry.

### 2 Materials and methods

Peaches (*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, and Chordas, 1982) determining a value of 2 kg (over-mature peaches) and their average diameter was 7 cm. The fresh peaches were cleaned, divided in halves and flesh was separated from the stone. Then, they were submitted to a juice extraction using a domestic appliance with the purpose of eliminating great part of the water content and its soluble solids.

The remaining tissue after juice extraction showed a moisture content of 23g/100g. It was contacted with ethanol (96 mL/100mL) using an ethanol/sample ratio of 200/100 or 350/100 or 500/100 and ethanol treatment was assayed at 20, 50 or 80° C using times of 15, 30 or 45 min for each temperature. The treatment was performed with a magnetic stirrer at 600 RPM (VELP Scientific, UE). Finally, ethanol was discarded and one portion of the remnant was dried at temperatures of 30, 50 or 70°C with air drying in a convective chamber. Another portion of the remnant was dried with microwave assistance at 40, 60 or 80°C. The two drying procedures were continued till the water activity of the fractions was lower than 0.6 and constant weight was attained. Water activities lower than 0.6 assure shelf stability of the fractions (Muggeridge and Clay, 2001).

The convective drying was performed in a pilot plant chamber equipped with trays. A high constant air-velocity of about 0.5 m/s was used. The trays contained an enough amount of material to form 1.0 cm depth layers.

Batch microwave processing was carried out in a microwave system ETHOS Plus (Milestone SRL, Sorisole, Italy) with a magnetron of 2450 MHz. The microwave used the ATC-400 system for continuous monitoring and control of the internal temperature. The optical sensor used was fitted in a teflon coated ceramic thermowell. The samples were distributed in plastic disposable food containers in layers of 1.0 cm deep. Then, they were placed inside a polypropylene container of cylindrical shape having a diameter of 27.9 cm diameter and a height of 19.7 cm. A 360° alternate movement was programmed for the container in the microwave cavity to avoid bending of the sensor connection during experiments while assuring a more homogeneous treatment of the samples. The container was covered with a perforated lid that served as support of the ceramic coated optical fiber. For each batch, the thermal sensor was placed in the center of one of the DF samples, and the temperature profiles were recorded during microwave processing.

All the fractions obtained were grounded and sifted in a mesh (sieve ASTM-USA, mesh 40). Particles with sizes smaller than 400  $\mu$ m were used for the following characterization. Samples were packed in Cryovac<sup>TM</sup>-bags (Cryovac CN 530 R film, GRACE, Argentina) which were sealed by heating. Bags were stored at –18 °C until usage.

### 2.1 DF fraction characterization

### 2.1.1 Yield

Yield was calculated as g of fraction obtained per 100g pressed (after juice extraction) raw material.

### 2.1.2 Physical and functional characterization

### 2.1.2.1 Water activity

Water activity was determined using an Aqualab CX-2 hygrometer (Decagon Devices Inc., Pullman, WA, USA) at 25°C. The determination was performed, at least, in duplicate.

### 2.1.2.2 Moisture content

Moisture was determined on  $\approx 0.5$  g samples by using infrared heating (Ohaus MB45 moisture analyzer Corporation, USA) till constant weight. The determination was performed, at least, in duplicate.

### 2.1.2.3 Apparent density and functional properties

Apparent density ( $\rho_a^{-1}$ ), hydration properties and oil holding capacity was evaluated according to de Escalada Pla et al. (2010). Briefly, apparent density ( $\rho_a^{-1}$ ) was determined by measuring the volume occupied by a weighed sample.

Hydration properties like *Swelling capacity (SC)*, *Water-holding capacity (WHC)* and *Water retention capacity (WRC)* were determined after hydrating an accurately weighed amount of DF fraction according to the following explanations.

**Swelling capacity (SC).** An accurately weighed dry sample ( $\approx 0.20$  g) was placed in a graduated conical tube. Around 10.0 mL of water was added and sample was hydrated for 18 h at 25°C-constant temperature. After this time, the volume of fiber product after hydration was measured. This assay was performed three times for each fraction. Swelling capacity was calculated as:

SC (ml/g) = Volume of sample after hydration / original sample weight

Water-holding capacity (WHC). An accurately weighed dry sample ( $\approx$  1.00 g) 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. After

freeze-drying, the residual dry weight was obtained. WHC was determined three times for each fraction.

WHC (g/g) = (Hydrated residue weight - dry residue weight) / dry residue weight

Water retention capacity (WRC) and retained water (RW). Water retention capacity and retained water were determined by hydration at 25°C for 18 h of an accurately weighed dry sample ( $\approx$  1.00 g) with 30.0 mL of water into a graduated conical tube. Centrifugation for 30 min at 2000 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 wet fiber retained was weighed (R+W<sub>2</sub>) and submitted to freeze-drying. The dried residue was then weighed (R). Then the parameters were calculated as:

WRC (g water/g dried residue) =  $W_2 / R$ 

RW (% of water retained) = ( $W_2$  / total water added) x 100

WHC represents water absorbed by DF without applying external forces, while WRC represents water retained by DF after centrifugal force was applied (de Escalada Pla et al., 2010).

Oil holding capacity (OHC) was measured by means of mixing  $\approx 0.20$  g of DF with sunflower oil ( $\approx 1.50$  g), and centrifuging after overnight contact of the system at room temperature. The supernatant was decanted and the sample was weighed, expressing OHC as g of oil absorbed/g dry sample.

All determinations were carried at least in triplicate for each fraction and mean values with corresponding standard deviation are reported.

### 2.2 Experimental design and statistical analysis

### 2.2.1 Experimental design

In order to find the main factors affecting yield and properties of fractions enriched in peach DF, a two level orthogonal design with four central points was formulated for 4 factors selected from preliminary studies. The factors were: time, temperature and ratio of ethanol to sample during ethanol treatment step and drying temperature. The range used for each factor were: for ethanol/ sample ratio, 200/100- 500/100 mL/g; for temperature of extraction, 20-80°C; for time of extraction, 15-45 min; for temperature of air drying, 30-70°C and for temperature of microwave assisted drying: 40-80°C (Table 1). Additionally, four central points were formulated with

the following values: for ethanol/sample ratio, 350/100; for temperature of extraction, 50°C; for time of extraction, 30 min; for temperature of air drying, 50°C and for micro-wave assisted drying, 60°C.

The design was performed for convective and for microwave drying and, in both cases, it was conducted in two blocks and central point was performed in quadruplicate for each block. A total of 24 systems were conducted for convective air drying and other 24 systems were performed for microwave drying.

### 2.2.2 Statistical analysis

Statistical analysis of the experimental design was performed according to Montgomery (2009). Pearson's product-moment correlations between the variables were also evaluated.

For the statistical analysis, the Statgraphics Centurion XV (02/15/06 V., 2007. Rockville, MD, USA) was used.

Independent Variables Density WRC OHC SC WHC Yield Time of drying Block Runs (g/100g) (min) (g/mL) (g/g) (g/g) (mL/g) (g/g) Α В С D 1 0 0 0 0 0 0.460±0.001 19.8±0.1 1.59±0.06 34±4 30±3 4.57 180 0 0 0 0.574±0.001 20±1 1.38±0.03 27±4 24±3 210 1 0 0 5.28 1 0 0 0.58±0.01 19.8±0.6 1.31±0.05 22±1 20±3 4.83 210 0 0 0 0 0 0.45±0.01 180 1 0 0 0 20.4±0.3  $1.7\pm0.1$ 27±2 23±3 4.60 1 1 1 1 1 1 0.508±0.008 19.9±0.7 1.55±0.09 25±2 23±2 4.62 140 1 2 1 -1 0.52±0.02 18.7±0.6 1.47±0.07 22.7±0.8 22.2±0.3 405 1 -1 5.75 1 3 1 -1 1 -1 0.47±0.01 25.1±0.9 1.678±0.004 31±2 31±1 6.00 405 1 4 1 -1 -1 1 0.63±0.02 20.7±0.5 1.6±0.2 26±3 27±3 7.27 160 1 5 -1 -1 0.49±0.02 19±1 1.62±0.04 27±3 25±2 4.69 405 1 1 1.08±0.05 22±4 1 6 -1 1 -1 1 0.71±0.02 16±1 22±2 4.82 150 0.58±0.04 19.0±0.4 1.3±0.1 43±3 4.97 160 1 7 -1 -1 1 1 43±3 1 8 -1 -1 -1 -1 0.62±0.01 17.8±0.6 1.14±0.06 34±3 37±3 5.74 405 2 0 0 0 0 0.5815±0.0007 1.4±0.1 29.4±0.9 27±3 250 0 16.4±0.8 5.12 2 0 0 0 0 0 0.61±0.02 17.2±0.7 1.5±0.2 32±1 29.1±0.7 5.08 250 2 0 0 0 0 0 0.66±0.03 17.6±0.8 1.4±0.1 29±7 22±3 5.20 270 2 0 0 0 0 0 0.61±0.02 17.9±0.9 1.4±0.1 35±2 34±2 5.00 220 2 9 0.54±0.02 20±1 1.53±0.09 23±2 28±2 300 1 1 1 -1 5.45 2 10 1 -1 1 0.76±0.02 14.8±0.1 1.3±0.2 16.3±0.3 20±1 6.23 205 1 2 11 1 -1 1 1  $0.617 \pm 0.008$ 20±1 1.8±0.1 32±2 30±1 4.88 190 2 12 1 -1 -1 -1 0.66±0.03 20.7±0.5 2.1±0.2 35.4±0.9 39±4 6.41 390 2 13 -1 1 1 1 0.61±0.01 15.6±0.7 1.7±0.1 20±1 20±1 4.45 190 2 14 -1 1 -1 -1 0.63±0.04 16±1 1.4±0.2 32±1 36±1 6.75 390 2 15 -1 -1 1 -1 0.669±0.008 18±1 1.4±0.1 27±2 28±1 5.50 390 2 16 -1 -1 -1 1 0.76±0.02 14.6±0.5 1.3±0.1 22.2±0.7 25.1±0.9 5.97 205

**Table 1:** Properties of fractions enriched in dietary fiber isolated from (*Prunus persica* L.) and air dried

A: Time of extraction; B: Temperature of extraction; C: Ethanol/Sample ratio and D: Temperature of air drying

A: 0 Coded= 30 min; +1 Coded= 45 min; -1 Coded= 15 min. B: 0 Coded= 50°C; +1 Coded= 80 °C; -1 Coded= 20 °C. C: 0 Coded= 350/100 mL/g

+1 Coded= 500/100 mL/g; -1 Coded= 200/100 mL/g. D: 0 Coded= 50°C; +1 Coded= 70 °C; -1 Coded= 30°C

### 3 Results and discussion

The treatment of plant tissues with ethanol followed by convective air drying has been previously assayed for the production of fractions enriched in dietary fiber (de Escalada Pla et al., 2007, 2010, 2012).

In the next section, a general comparison of the effect observed on DF properties in relation to the application of convective or microwave drying will be discussed.

### 3.1 General comparison between different drying techniques

Tables 1 and 2 show the experimental results of the yield, apparent density, WRC, SC, WHC, OHC and drying time, observed for the fractions obtained through the two drying techniques assayed .

Table 3 and 4 summarize significant effects of the processing factors on peach DF properties when convective

Table 2: Properties of fractions enriched in dietary fiber isolated from (Prunus persica L.) and microwave dried

| Block | Runs | Independent<br>Variables |    |    |    | Density         | WRC        | онс         | SC         | WHC        | Yield    | Time of      |
|-------|------|--------------------------|----|----|----|-----------------|------------|-------------|------------|------------|----------|--------------|
|       |      | Α                        | В  | С  | D  | (g/mL)          | (g/g)      | (g/g)       | (mL/g)     | (g/g)      | (g/100g) | drying (min) |
| 1     | 0    | 0                        | 0  | 0  | 0  | 0.55 ± 0.03     | 15.6 ± 0.5 | 1.43 ± 0.02 | 14.6 ± 0.7 | 14 ± 1     | 4.50     | 35           |
| 1     | 0    | 0                        | 0  | 0  | 0  | $0.61 \pm 0.01$ | 13.0 ± 0.5 | 1.20 ± 0.06 | 12 ± 1     | 11 ± 2     | 4.74     | 35           |
| 1     | 0    | 0                        | 0  | 0  | 0  | 0.51 ± 0.04     | 17.6 ± 0.3 | 1.67 ± 0.02 | 18 ± 1     | 19.1 ± 0.4 | 5.11     | 35           |
| 1     | 0    | 0                        | 0  | 0  | 0  | 0.55 ± 0.04     | 14.4 ± 0.2 | 1.60 ± 0.08 | 14 ± 1     | 14.0 ± 0.8 | 4.38     | 30           |
| 1     | 1    | 1                        | 1  | 1  | 1  | 0.49 ± 0.03     | 16.7 ± 0.4 | 1.724±0.007 | 17.6 ± 0.4 | 17.3 ± 0.4 | 4.44     | 22           |
| 1     | 2    | 1                        | 1  | -1 | -1 | 0.63 ± 0.03     | 16.2 ± 0.1 | 1.27 ± 0.08 | 16 ± 1     | 17 ± 1     | 5.40     | 210          |
| 1     | 3    | 1                        | -1 | 1  | -1 | 0.6 ± 0.1       | 19 ± 1     | 1.4 ± 0.1   | 29 ± 3     | 29 ± 1     | 5.71     | 210          |
| 1     | 4    | 1                        | -1 | -1 | 1  | 0.65 ± 0.02     | 8.6 ± 0.3  | 1.30 ± 0.08 | 6.5 ± 0.4  | 9 ± 1      | 5.78     | 22           |
| 1     | 5    | -1                       | 1  | 1  | -1 | $0.44 \pm 0.01$ | 20.3 ± 0.9 | 2.0 ± 0.1   | 32.4 ± 0.8 | 29.3 ± 0.7 | 5.89     | 210          |
| 1     | 6    | -1                       | 1  | -1 | 1  | 0.58 ± 0.02     | 12.5 ± 0.9 | 1.48 ± 0.05 | 12.0 ± 0.8 | 12 ± 1     | 5.68     | 26           |
| 1     | 7    | -1                       | -1 | 1  | 1  | 0.51 ± 0.02     | 19.7 ± 0.2 | 1.66 ± 0.06 | 27 ± 2     | 23 ± 2     | 5.58     | 22           |
| 1     | 8    | -1                       | -1 | -1 | -1 | 0.62 ± 0.06     | 19 ± 1     | 1.21 ± 0.06 | 26 ± 2     | 25 ± 2     | 6.56     | 210          |
| 2     | 0    | 0                        | 0  | 0  | 0  | 0.65 ± 0.05     | 22 ± 1     | 1.20 ± 0.01 | 21.1 ± 0.7 | 19 ± 1     | 5.30     | 55           |
| 2     | 0    | 0                        | 0  | 0  | 0  | 0.62 ± 0.02     | 22 ± 1     | 1.44 ± 0.06 | 29 ± 2     | 27 ± 1     | 5.57     | 50           |
| 2     | 0    | 0                        | 0  | 0  | 0  | 0.644 ± 0.009   | 22.7 ± 0.9 | 1.31 ± 0.09 | 20 ± 1     | 18 ± 1     | 4.60     | 50           |
| 2     | 0    | 0                        | 0  | 0  | 0  | 0.6610±0.0002   | 19.3 ± 0.4 | 1.20 ± 0.06 | 21 ± 2     | 21 ± 2     | 4.90     | 50           |
| 2     | 9    | 1                        | 1  | 1  | -1 | 0.7 ± 0.2       | 24 ± 3     | 1.53 ± 0.06 | 20 ± 3     | 18 ± 1     | 4.86     | 260          |
| 2     | 10   | 1                        | 1  | -1 | 1  | 0.71 ± 0.02     | 12.2 ± 0.2 | 1.44 ± 0.05 | 11 ± 1     | 12 ± 1     | 5.83     | 38           |
| 2     | 11   | 1                        | -1 | 1  | 1  | 0.578 ± 0.006   | 19.9 ± 0.8 | 1.67 ± 0.07 | 20.8 ± 0.4 | 21 ± 1     | 5.96     | 34           |
| 2     | 12   | 1                        | -1 | -1 | -1 | 0.66 ± 0.02     | 21.4 ± 0.5 | 1.4 ± 0.1   | 32.5 ± 0.7 | 34 ± 1     | 5.86     | 275          |
| 2     | 13   | -1                       | 1  | 1  | 1  | 0.669 ± 0.003   | 18.7 ± 0.4 | 1.45 ± 0.05 | 16 ± 1     | 17 ± 2     | 5.19     | 38           |
| 2     | 14   | -1                       | 1  | -1 | -1 | 0.76 ± 0.02     | 21 ± 2     | 1.20 ±0.05  | 26 ± 2     | 28 ± 2     | 5.51     | 275          |
| 2     | 15   | -1                       | -1 | 1  | -1 | 0.599 ± 0,004   | 23.8 ± 0.7 | 1.66 ± 0.06 | 36 ± 2     | 36 ± 2     | 5.43     | 260          |
| 2     | 16   | -1                       | -1 | -1 | 1  | 0.73 ± 0.02     | 11.2 ± 0.4 | 1.22 ± 0.02 | 10 ± 1     | 11 ± 1     | 5.76     | 38           |

A: Time of extraction; B: Temperature of extraction; C: Ethanol/Sample ratio and D: Temperature of microwave drying A: 0 Coded= 30 min; +1 Coded= 45 min; -1 Coded= 15 min. B: 0 Coded= 50°C; +1 Coded= 80 °C; -1 Coded= 20 °C. C: 0 Coded= 350/100 L/g

+1 Coded= 500/100 mL/g; -1 Coded= 200/100 mL/g. D: 0 Coded= 60°C; +1 Coded= 80 °C; -1 Coded= 40°C

|                             | Density<br>(g/mL) | WRC<br>(g/g) | OHC<br>(g/g) | SC<br>(mL/g) | WHC<br>(g/g) | Yield<br>(g/100g) |
|-----------------------------|-------------------|--------------|--------------|--------------|--------------|-------------------|
| Independent term            | 0.597             | 18.568       | 1.493        | 28.282       | 28.367       | 5.383             |
| A:Time of extraction        | -0.023            | 1.423**      | 0.117*       | -1.070       | -1.059       | 0.233**           |
| B:Temperature of extraction | -0.016            | -0.948**     | -0.045       | -3.862**     | -3.941*      | -0.247**          |
| C:Ethanol for extraction    | -0.051*           | 0.987**      | 0.074        | 1.206        | -0.057       | -0.523**          |
| D:Temperature of drying     | 0.036*            | -0.919**     | -0.047       | -1.647       | -2.317       | -0.192            |
| AB                          | 0.008             | -0.704**     | -0.115*      | -0.838       | -0.277       | -0.065            |
| AC                          | -0.003            | 0.198        | -0.063       | 0.268        | 0.443        | -0.065            |
| AD                          | 0.004             | -0.210       | -0.022       | 0.059        | -0.154       | 0.117             |
| BC                          | -0.009            | 0.013        | 0.063        | -0.922       | -0.605       | -0.019            |
| BD                          | 0.015             | 0.016        | -0.009       | -1.008       | -1.022       | -0.122            |
| CD                          | -0.017            | -0.038       | 0.059        | 3.099        | 2.592*       | -0.148            |
| R2                          | 80.71             | 95.80        | 79.42        | 61.58        | 61.10        | 63.51             |
| Lack of fit                 | 0.626             | 0.186        | 0.464        | 0.180        | 0.120        | 0.003             |

### Table 3: Coefficients of regression and ANOVA for properties of DF obtained with convective air drying

\* p< 0.05, \*\*p< 0.01

Table 4: Coefficients of regression and ANOVA for properties of DF obtained with microwave drying

|                             | Density<br>(g/mL) | WRC<br>(g/g) | OHC<br>(g/g) | SC<br>(mL/g) | WHC<br>(g/g) | Yield<br>(g/100g) |
|-----------------------------|-------------------|--------------|--------------|--------------|--------------|-------------------|
| Independent term            | 0.613             | 17.950       | 1.444        | 20.354       | 20.071       | 5.356             |
| A:Time of extraction        | 0.007             | -0.512       | -0.009       | -2.000       | -1.500       | -0.110            |
| B:Temperature of extraction | 0.002             | -0.063       | 0.036        | -2.300       | -2.338       | -0.240            |
| C:Ethanol for extraction    | -0.047**          | 2.500**      | 0.161**      | 3.675**      | 2.663        | -0.208            |
| D:Temperature of drying     | -0.006            | -2.825**     | 0.017        | -6.063**     | -5.875**     | -0.063            |
| AB                          | 0.003             | 0.088        | -0.012       | -0.725       | -1.250       | -0.108            |
| AC                          | 0.012             | 0.150        | -0.047       | -1.000       | -1.000       | -0.030            |
| AD                          | -0.015            | -0.075       | 0.050        | 0.863        | 1.038        | 0.085             |
| BC                          | -0.001            | -0.275       | 0.003        | -1.050       | -1.088       | -0.048            |
| BD                          | -0.004            | 0.150        | -0.005       | 1.338        | 1.625        | -0.003            |
| CD                          | -0.006            | 1.313        | -0.028       | 1.563        | 1.625        | -0.028            |
| R <sup>2</sup>              | 76.42             | 85.47        | 57.41        | 78.48        | 79.70        | 31.12             |
| Lack of fit                 | 0.044             | 0.172        | 0.300        | 0.082        | 0.186        | 0.0383            |

\*p< 0.05, \*\*p< 0.01

air drying and microwave assisted drying were used. It can be observed that the convective drying demanded a time of process that ranged between 140 and 405 min while for microwave assisted drying, processing times ranged between 22 and 275 min to attain water activities lower than 0.6.

As can be observed in Tables 1 and 2, shorter drying times where observed in the present research when microwaves were used. Maskan (2001) reported that microwave assisted heating produced a reduction of drying times of kiwifruits slices by 89-40% in comparison to hot air drying. Microwave heating takes place in dielectric materials such as vegetables tissues due to the polarization effect of electromagnetic radiation at frequencies between 300 MHz and 300 GHz. The heating by microwave is a consequence of the conversion of the electromagnetic energy in heat by selective absorption and dissipation. As microwaves can transfer energy through out the volume of material, this transfer of energy offers advantages such as processing time reduction and overall quality enhancing when compared to thermal conventional processing (Latorre et al., 2013).

Processes applied led to obtain yields ranging between 4.38 and 7.27 g/100g. Due to negative effects observed for ethanol/sample ratio and extraction temperature when using convective drying (Table 3), it could be inferred that the increase of these variables tended to render lower DF fraction yield. No significant effects were observed for yield when microwave was used. Nevertheless, it must be considered that, in both cases, convective and microwave drying, the model applied did not describe adequately the yield behavior. Low value for lack of fit (Tables 4 and 5) and also for  $R^2$  (Table 4) were observed. For this reason, values for the coefficients are shown on Table 3 and 4 but yield will not be analyzed in the following section.

On the other hand, it was observed that conditions (time and/or temperature) of the ethanol extraction step previous to drying presented significant effects on some properties when a subsequent convective drying was employed, while no effects of these conditions on hydration properties or oil holding capacity, were observed when microwave drying was used. The effect of the ethanol/sample ratio and of the temperature of drying on oil holding capacity and hydration properties was mainly observed on samples dried by microwave technique (Table 3 and 4).

## **3.2** Analyses of the effect of different factors involved in ethanol treatment and of drying temperature on the DF properties

According to the experimental design, several runs were carried out in order to evaluate the effect of the ethanol treatment and the drying temperature on the properties studied. Coefficients of regression were determined and ANOVA was performed for the experimental data to identify the effect of different factors (Table 3 and Table 4).

Block effects were observed only for density and WRC. These block effects may be attributed to environmental conditions. As the drying mechanisms involve water transfer from DF to surrounding air, the air relative humidity conditions have an important effect on driving force during mass transfer and, probably, on some of the properties of the final product.

It can be observed in Table 3 that ethanol/sample ratio and temperature of drying significantly (p<0.05) affected DF density when air drying was used. The ethanol/sample ratio had a negative effect, while temperature of drying presented a positive effect (p<0.05) showing that higher ethanol/sample ratios and lower drying temperatures could produce DF fractions with lower densities (Table 3). Similar results were observed for ethanol/sample ratio when microwave drying was used (Table 4). It is important to remark that although R<sup>2</sup> is high, the lack of fit is not significant for density when microwave drying was used. Density depends on the water content, on the physicochemical characteristics of the solids and on the proportion of air volume which is affected by the method of drying and by the collapse of the matrix (Koc, et al., 2008). De Escalada Pla et al. (2010) reported lower values of density for quince DF when an ethanol treatment previous to drying was applied instead of an aqueous treatment.

All the factors affected WRC significantly (p<0.01) when DF fractions were air dried. In this case, time of extraction presented the higher positive effect. Ethanol/ sample ratio also showed a positive effect (p<0.01) and, finally, both temperatures of extraction and of drying had a negative effect (p<0.01). Higher temperatures, produced DF fractions with lower WRC; while, longer times of extraction and higher ethanol/sample ratios would be recommended to increase WRC. Antagonistic interaction between time and temperature of extraction was also observed (p<0.01) for air dried DF (Table 3). When microwave was used, only the effects of ethanol/sample ratio

and temperature of drying could be detected (p<0.01) (Table 4). The former had a positive effect while the temperature of drying had a negative effect (p<0.01). In other words, to obtain DF fractions with enhanced WRC when microwaves were used, higher ethanol/sample ratios and lower drying temperature should be recommended.

For air dried DF, it was observed a negative effect (p<0.01; p<0.05) of the temperature of extraction on SC and WHF and, for WHC, a positive (p<0.05) effect of the interaction between the ethanol/sample ratio and the drying temperature although each factor did not exert a significant effect by itself (Table 3). When microwave was applied, temperature of drying affected negatively (p<0.01) SC and WHC (Table 4). Ethanol/sample ratio affected positively SC (p<0.01) and no interactions were detected (Table 4).

For the OHC, the time of extraction exerted a positive (p<0.05) effect and an antagonistic interaction between time and temperature of extraction was observed when air drying was applied (Table 3). When microwaves were used, ethanol/sample ratio was the only factor that affected OHC (p<0.01). OHC for microwave assisted drying was the property that fit to the proposed model with the lower  $R^2$ .

In general, hydration properties and also OHC showed increased values when higher ethanol/sample ratios and lower temperatures of microwave drying were used (Table 4). The direct treatment of a vegetable sample with hot ethanol produces the isolation of the cell wall material (Renard, 2005). Nevertheless, free glucose or low molecular soluble compounds could still remain in the product (de Escalada Pla et al., 2010). Then, matrix damage could occur if drying is performed at high temperatures, due to collapse of the pores or shrinkage of the tissue, in the presence of low molecular saccharides (Gerschenson et al., 1981; Guillon et al., 1998). Possibly, higher ethanol/ sample ratios could help to the elimination of water and low molecular soluble saccharides while, simultaneously, lower drying temperatures could reduce matrix collapse. In this way, apparent density tended to decrease and, simultaneously, hydration properties as well as OHC tended to increase (Table 4). Ghanem et. al. (2012) reported that, in citrus, volume decrease (shrinkage) did not depend on the peel cultivar variety and on the microwave drying level but on the quantity of evaporated moisture.

It is important to remark that values reported for the different properties and yield when convection drying was used (Table 3), were similar to those reported by de Escalada Pla et al (2012) for peach peel or pulp treated with ethanol (boiling ethanol/sample ratio: 350/100 mL/g; 15 min of stirring) and dehydrated at 30°C during 7 h by

air convection. WRC and OHC resulted in all cases slightly higher than values reported by Grigelmo-Miguel et al. (1999) for DF obtained from peach bagasse.

### 3.3 Analyses of the correlations among DF properties

The Pearson product moment correlation coefficients range from -1 (negative dependence) to +1 (positive dependence), and measure the strength of the linear relationship between the variables. Table 5 and Table 6 report the results in the form of the coefficients for each pair of variables. It is also shown, in parentheses, the statistical significance of the estimated correlations expressed through the p value.

For both drying procedures, practically a linear correlation was detected for WHC and SC as expected because both properties are strongly linked by their way of determination and calculation.

It can be observed in Table 5 the Pearson product moment correlation for air dried DF. A positive correlation was observed between WRC and OHC. The WRC was also negatively correlated with the apparent density.

In Table 6 Pearson product moment are reported for samples dried through microwaves. In this case, significant and positive correlations between all hydration properties were detected and also a negative one between OHC and apparent density.

Many authors have reported a relationship between WHC and OHC with the bulk density or the specific volume (Guillon and Champ, 2000; Prakongpan et al. 2002; Chau et al., 2004). For quince DF, de Escalada Pla et al. (2010) reported that OHC essentially depended on density and the microstructural characteristics of the fiber powders.

### **4** Conclusions

The process studied for obtaining fractions enriched in DF resulted in values of yield ranging from 4.57 to 7.27 g/100g for convection and 4.38 to 6.56 g/100 for microwave drying. In general, it was observed that the drying time necessary for attaining a water activity that assures shelf stability, was lower for the microwave drying procedure.

Processing conditions (time and temperature) for the ethanol treatment step presented significant effects on DF properties when a subsequent convective drying was employed. The effect of the ethanol/sample ratio and of the temperature of drying on oil holding capacity and

|                   | Density<br>(g/mL)    | OHC<br>(g/g)       | SC<br>(mL/g)         | WHC<br>(g/g)         | WRC<br>(g/g)         |
|-------------------|----------------------|--------------------|----------------------|----------------------|----------------------|
| Density<br>(g/mL) | 1                    | -0.3637            | -0.2641              | -0.0018              | -0.6642<br>(p<0.001) |
| OHC<br>(g/g)      | -0.3637              | 1                  | 0.1942               | 0.1260               | 0.5249<br>(p<0.05)   |
| SC<br>(mL/g)      | -0.2641              | 0.1942             | 1                    | 0.8991<br>(p<0.0001) | 0.3657               |
| WHC<br>(g/g)      | -0.0018              | 0.1260             | 0.8991<br>(p<0.0001) | 1                    | 0.2481               |
| WRC<br>(g/g)      | -0.6642<br>(p<0.001) | 0.5249<br>(p<0.05) | 0.3657               | 0.2481               | 1                    |

Table 5: Pearson product moment coefficients between variables for peach DF obtained with convective air drying

Significant correlations are highlighted in gray. In parentheses, the statistical significance of the estimated correlations is reported

Table 6: Pearson product moment coefficients between variables for peach DF obtained with microwave drying

|                   | Density              | ОНС                   | SC                   | WHC                  | WRC                  |
|-------------------|----------------------|-----------------------|----------------------|----------------------|----------------------|
|                   | (g/mL)               | (g/g)                 | (mL/g)               | (g/g)                | (g/g)                |
| Density<br>(g/mL) | 1                    | -0.7538<br>(p<0.0001) | -0.2033              | -0.1177              | -0.0186              |
| OHC<br>(g/g)      | -0.7538<br>(p<0.0001 | 1                     | 0.3170               | 0.2653               | 0.1952               |
| SC<br>(mL/g)      | -0.2033              | 0.3170                | 1                    | 0.9796<br>(p<0.0001) | 0.8157<br>(p<0.0001) |
| WHC<br>(g/g)      | -0.1177              | 0.2653                | 0.9796<br>(p<0.0001) | 1                    | 0.7651<br>(p<0.0001) |
| WRC<br>(g/g)      | -0.0186              | 0.1952                | 0.8157<br>(p<0.0001) | 0.7651<br>(p<0.0001) | 1                    |

Significant correlations are highlighted in gray. In parentheses, the statistical significance of the estimated correlations is reported

hydration properties was mainly detected on samples dried by microwave technique.

Higher ethanol/sample ratios and lower drying temperatures could produce air dried DF fractions with lower densities. Similar results were observed with microwave drying, but only ethanol/ sample ratio effect was detected.

In general, when microwave assisted drying was used, enhanced hydration properties and also OHC could be obtained using higher ethanol/sample ratios and reducing temperature of drying.

Results obtained showed that the drying technique is a key factor in relation to the characteristics of fractions enriched in dietary fiber and that microwave drying allowed to obtain fractions with functional properties that can be modulated through the use of different relations of ethanol to sample during treatment and with the temperature of drying.

The results provide insight into the effect of processing of plant residues on the properties of DF fractions obtained and on their potential performance as ingredients or additives for the food industry.

Acknowledgements: This study was financially supported by University of Buenos Aires (UBACyT 20020100100726 and 20020130100550BA/2014-2017), National Agency of Scientific and Technical Research (PICT 2008-38239 and 2013-2088) and CONICET (PIP 11220090100531 and 11220120100507/2013-2015)

### References

- Arslan R., Özcan M.M., Study the effect of sun, oven and microwave drying on quality of onion slices, LWT-Food Sci. Technol., 2010, 43, 1121-1127
- [2] Chau C.F., Wang Y.T., Wen Y.L., Comparison of the characteristics, functional properties and in vitro hypoglycemic effects of various carrot insoluble fiber-rich fractions, LWT-Food Sci. Technol., 2004, 37, 155-160
- [3] de Escalada Pla M., González P., Sette P., Portillo F., Rojas A.M., Gerschenson L.N., Effect of processing on physico-chemical characteristics of dietary fiber concentrates obtained from peach (Prunus persica L.) peel and pulp, Food Res. Int., 2012, 49, 184-192
- [4] de Escalada Pla M., Uribe M., Fissore E., Gerschenson L.N., Rojas A.M., Influence of the isolation procedure on the characteristics of fiber-rich products obtained from quince wastes, J.Food Eng., 2010, 96, 239-248
- [5] de Escalada Pla M., Ponce N.M., Stortz C.A., Gerschenson L.N., Rojas A.M., Composition and functional properties of enriched fibre products obtained from pumpkin (Cucurbita moschata Duchesne ex Poiret), LWT-Food Sci. Technol., 2007, 40, 1176–1185
- [6] Food and Nutrition Board, Institute of Medicine, Dietary reference intakes for energy, carbohydrate, fiber, fat, fatty acids, cholesterol, protein and amino acids, National Academy Press, Washington, DC, 2005
- [7] Gerschenson L.N., Bartholomai G:, Chirife J., Structural collapse and volatile retention during heating and rehumidification of freeze dried tomato juice, J.Food Sci., 1981, 46 (5), 1552-1556
- [8] Ghanema N., Mihoubib D., Kechaoua N., Mihoubic N., Microwave dehydration of three citrus peel cultivars: Effect on water and oil retention capacities, color, shrinkage and total phenols content, Ind. Crop.Prod., 2012, 40, 167- 177
- [9] Goñi I., Díaz-Rubio M., Pérez-Jiménez J., Saura-Calixto F., Towards an update methodology for measurement of dietary fiber, including associate polyphenols in food and beverages. Food Res. Int., 2009, 42, 840-846.
- [10] Grigelmo-Miguel N., Martin-Belloso O., Comparison of dietary fibre from byproducts of processing fruits and greens and from cereals, LWT-Food Sci. Technol., 1999, 32, 503-508
- [11] Guillon F., Auffret A., Robertson J., Thibault J., Barry J., Relationships between physical characteristics of sugar-beet fibre

and its fermentability by human faecal flora, Carbohyd. Polym., 1998, 37, 185-197

- [12] Guillon F., Champ M., Structural and physical properties of dietary fibers, and consequences of processing on human physiology, Food Res. Int., 2000, 33, 233-245
- [13] Kader A., Heintz C., Chordas A., Postharvest quality of fresh and canned Clingstone peaches as influenced by genotypes and maturity at harvest, J.Am. Soc. Hortic. Sci., 1982, 107(6), 947-951
- [14] Koç B., Eren I., Ertekin F., Modelling bulk density, porosity and shrinkage of quince during drying: The effect of drying method, J.Food Eng., 2008, 85, 340-349
- [15] Latorre M.E., Escalada Pla M., Rojas A. M., Gerschenson L.N.
   Blanching of red beet (Beta vulgaris L. var. conditiva) root.
   Effect of hot water or microwave radiation on cell wall characteristics. LWT–Food Sci. Technol., 2000, 50(1), 193-203
- [16] Maskan M., Drying, shrinkage and rehydration characteristics of kiwifruits during hot air and microwave drying, J.Food Eng., 2001, 48: 177-182
- [17] Montgomery D., Design and Analysis of experiments, John Wiley and Sons, Chichester, West Sussex, UK, 2009
- [18] Muggeridge M., Clay M., Introduction, In: Peter KV (Ed.), Handbook of Herbs and Spices. Woodhead Publishing Limited, Cambridge, 2001
- [19] Murthy G., Prasad S., A completely coupled model for microwave heating of foods in microwave oven, An ASAE Meeting Presentation Paper Number, 2005, https://www. researchgate.net/publication/271438553\_A\_Completely\_ Coupled\_Model\_for\_Microwave\_Heating\_of\_Foods\_in\_ Microwave\_Oven
- [20] NOAA Research. U.S. Department of Commerce. National Oceanic & Atmospheric Administration, Trends in Atmospheric Carbon Dioxide, 2013, http://www.esrl.noaa.gov/gmd/ccgg/ trends/ (accessed on July 1, 2013)
- [21] Prakongpan T., Nitithamyong A., Luangpituksa P., Extraction and application of dietary fiber and cellulose from pineapple cores, J.Food Sci., 2002, 67(4), 1308-1313
- [22] Renard C., Variability in cell wall preparations: quantification and comparison of common methods, Carbohyd. Polym., 2005, 60, 515-522
- [23] Spinner J., Food companies need to watch their waste, 2013, http://www.foodproductiondaily.com/Processing/ Food-companies-need-to-watch-their-waste