

# Effect of air-drying temperature on physico-chemical properties of dietary fibre and antioxidant capacity of orange (*Citrus aurantium* v. Canoneta) by-products

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## Abstract

Dehydration promoted important modifications affecting both the physico-chemical properties of dietary fibre (DF) and the antioxidant capacity of orange by-products (peel and pulp remaining after juice extraction). The significance of such changes was largely dependent on the air-drying temperature used (from 30 °C to 90 °C). The major modifications on the DF components were observed when either extended drying periods, i.e. at lower temperatures, or elevated drying temperatures were applied. Dehydration around 50–60 °C apparently promoted the minor disruption of cell wall polymers, in particular of pectic substances. Pulp samples exhibited higher values of swelling (SW) and fat adsorption capacity (FAC) than those derived from orange peel. Although, significant decreases in water retention capacity (WRC), FAC and solubility values were detected for both by-products as the air-drying temperature increased. The antioxidant capacity associated to dehydrated citrus by-products was significantly higher for orange peel than for pulp samples. In general, the by-products studied proved to be quite resistant to the different heat treatments applied within the range of 40–70 °C. In overall, the study shows that, in order to preserve either the DF quality and/or the antioxidant capacity, air-drying temperature should be controlled since both types of compounds, DF components and antioxidants, might be degraded or modified either when extended drying periods and/or high drying temperatures are applied.

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**Keywords:** Orange by-products; *Citrus aurantium*; Air-drying temperature; Dietary fibre; Functional properties; Antioxidant capacity

## 1. Introduction

For the last decades, the demand for appropriate nutritional and health standards has increased considerably. This has been characterized by rising costs and often decreasing the availability of raw materials together with much concern about environmental pollution. Consequently there is a considerable emphasis on the recovery, recycling and upgrading of wastes. This is particularly valid for the food and food processing industry in which wastes, effluents, residues, and by-products can be recovered and can often be upgraded to higher value and useful products

(Laufenberg, Kunz, & Nystroem, 2003; Reddy & Yang, 2005).

Spain is one of the major producers and exporters of citrus fruits. For some time, the food industry has shown a special interest in finding uses for citrus industry by-products (Larrea, Chang, & Martinez-Bustos, 2005a). During the citrus juice extraction process, thousands of tons of by-products are produced. These are mainly used for animal feeds, although, due to their high fibre content, they could represent an interesting source of dietary fibre (DF) (Lario et al., 2004; Mandalari et al., 2006).

DF acts as a bulking agent, normalizing intestinal motility and preventing diverticular disease. Considerable attention has also been focused on the incidence of a number of non-infectious diseases common in civilized societies, such

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as coronary heart disease, which could be attributed to a low DF intake. Some types of DF may also be important in reducing colonic cancer, in lowering serum cholesterol levels and in preventing hyperglycemias in diabetic patients (Nawirska & Kwasniewska, 2005). For this reason, in recent years diverse products containing important amounts of DF have been developed (Larrea, Chang, & Martinez-Bustos, 2005b).

Therefore, the importance of DF in the diet leads to a search for new sources of DF which can be used as food ingredients (Chau & Huang, 2003; Rodríguez, Jiménez, Fernández-Bolaños, Guillén, & Heredia, 2006). The development of new citrus products might be interesting in order to promote their consumption according to the current tendencies. The interest of orange by-products derived from the juice extraction and agricultural industries is mainly based on its potential use as a DF sources.

DF from cereals are more frequently used than DF from fruits. However, fruit fibres are being considered of higher quality due to a better balance of soluble and insoluble DF content and, also, due to their higher water and oil holding capacities (Larrauri, 1999). Concentrates obtained from fruit and vegetables after dehydration might contain important amounts of total DF (25–60 g/100 g dry matter [DM]) and better soluble–insoluble DF ratios than cereal brans (Garau, Simal, Femenia, & Rosselló, 2006).

An important problem in a wide range of food industries is the rate of oxidation of different food products. This phenomenon can be avoided or retarded by means of antioxidants, either synthetic or natural ones. Actually, the use of synthetic antioxidants in the food industry is under great consideration mainly due to toxicological reasons (Rehman, 2006), and the interest in the natural compounds is steadily increasing (Louli, Ragoussis, & Magoulas, 2004). In this sense, citrus fruit by-products could be interesting not only for its important fibre content but also because of its antioxidant capacity (Kang, Chawla, Jo, Kwon, & Byun, 2006; Rehman, 2006). They have a high fibre and vitamin contents as well as other associated bioactive compounds such as flavonoids and terpenes which exhibit interesting antioxidant properties (Lario et al., 2004). It has been reported that different phenolic compounds exhibit antioxidant activity and some authors have also claimed that certain parts of what is considered as DF might also exert antioxidant effects (Gorinstein et al., 2001; Lario et al., 2004).

Dietary fibres are desirable not only for their nutritional aspects but also for their functional and technological properties (Schieber, Stintzing, & Carle, 2001; Thebaudin, Lefebvre, Harrington, & Bourgeois, 1997). It is important to be aware of the processing history of fibre concentrates, in particular on the ability of the fibre matrix to maintain its physical properties after being processed (Femenia, Lefebvre, Thebaudin, Robertson, & Bourgeois, 1997).

Drying has become a widely used way of food processing allowing the extension of the shelf-life of fruits and vegetables. However, processing may cause irreversible

modifications to the cell wall polysaccharides, affecting their original structure. This may promote important changes in the proposed physiological and pharmacological properties of these polymers (Femenia, 2007). Therefore, the final quality of the dried by-products would be determined by the structural and compositional modifications which might have occurred during the drying treatment (Femenia, García-Pascual, Simal, & Rosselló, 2003).

The main objectives of the present study were, on the one hand, to evaluate the effects of dehydration on the cell wall composition and the functional properties of DF obtained from two by-products derived from orange fruit processing (orange peel and orange pulp remaining after juice extraction), and, on the other hand, to assess the influence of the air-drying temperature on their antioxidant activity.

## 2. Materials and methods

### 2.1. Material

Fresh oranges (*Citrus aurantium* v. Canoneta) were obtained from different cultivars of the island of Majorca (Spain). *Citrus aurantium* v. Canoneta is commonly used for juice production. Oranges hand-picked from the trees were washed, half cut and after the juice extraction, the peel and the remaining pulp were separated. The main morphological and physico-chemical parameters corresponding to the *Citrus aurantium* v. Canoneta are shown in Table 1.

### 2.2. Drying

Samples of both citrus by-products, peel and pulp, were dried in a pilot-scale hot air drier (Femenia et al., 2003). Samples were dried by convection drying at temperatures of 30 °C, 40 °C, 50 °C, 60 °C, 70 °C, 80 °C and 90 °C.

Table 1  
Morphological and physico-chemical features of the *Citrus aurantium* v. Canoneta

Parameter	Value	Units
Diameter	6.6 ± 0.4	cm
Weight	184 ± 38	g
Skin thickness	4.6 ± 0.8	mm
Color		
$L^*$	69.7 ± 2.5	–
$a^*$	16.3 ± 3.1	–
$b^*$	69.5 ± 3.1	–
% Skin	39.5 ± 7.3	g/100 g
% Pulp	34.0 ± 7.7	g/100 g
Moisture	86.3 ± 0.6	g/100 g
pH	4.06 ± 0.31	–
Dietary fibre	6.25 ± 0.48	g/100 g
Soluble sugars	5.62 ± 0.32	g/100 g
Ash	0.52 ± 0.20	g/100 g
Protein	1.08 ± 0.15	g/100 g
Fat	0.23 ± 0.05	g/100 g

The air flow rate was of 2 m/s in all experiments. Drying was performed till a constant moisture content for both type of samples, 0.12 and 0.11 g H<sub>2</sub>O/100 g DM for peel and pulp samples, respectively. A freeze-dried sample of each by-product was taken as a reference when needed.

### 2.3. Color

Color values of dried samples were measured using a Minolta CR 300, C.M. 2002 Spectrocolorimeter with the specular component included, C illuminant, and an observer with an angle of 2°.

The color values were expressed using CIELab\* coordinates where  $L^*$  represents the luminosity (0 = black; 100 = white),  $a^*$  the redness ( $a^* > 0$ ) or greenness ( $a^* < 0$ ) and  $b^*$  the blueness ( $b^* > 0$ ) or yellowness ( $b^* < 0$ ). A freeze dried sample which did not show any apparent browning sign was taken as a reference. The changes in lightness of each color parameters were calculated as follows:

$$\Delta L = L - L_0$$

whereas the total color difference ( $\Delta E$ ) was then determined using the following equation:

$$\Delta E = \sqrt{(L - L_0)^2 + (a - a_0)^2 + (b - b_0)^2}$$

The subscript "0" in both equations refers to the freeze dried sample for each orange by-product (either peel or pulp).

### 2.4. Alcohol insoluble residues

Alcohol insoluble residues (AIRs) from fresh and dehydrated samples of both by-products were prepared in order to evaluate the physico-chemical properties of the DF components. AIRs were obtained by immersing the samples, either of orange peel or pulp, in boiling ethanol (final concentration 85% (v/v) aq.) as described in Femenia, Robertson, Waldron, and Selvendran (1998a). Prior to further analysis, the AIRs were milled using a laboratory type grain mill and passed through a 0.5 mm aperture sieve.

### 2.5. Analytical methods

Moisture, lipids, soluble sugars, protein, lignin and ash analyses were performed in triplicate. Average and standard deviation values are shown in the tables.

**Moisture.** The moisture content was obtained according to the AOAC method no. 934.06 (AOAC, 1990).

**Water activity.** The water activity was measured at 25 °C by using an electric hygrometer NOVASINA thermoconstanter TH200.

**Protein.** Total nitrogen content was determined by the Kjeldahl method using Tecator equipment (digester model 2020 and Distillation and Tritation Kjeltex 1035/38 system). Protein content was estimated by multiplying the nitrogen value by 6.25.

**Soluble sugars.** Soluble sugars were measured colorimetrically using the anthrone reagent as in Femenia, Rosselló, Mulet, and Cañellas (1995).

**Ash.** Ash contents of dehydrated samples were gravimetrically determined by overnight heating at 550 °C, according to the AOAC method no. 945.46 (AOAC, 1997).

**Lipids.** Total content of lipids was determined gravimetrically by extraction with diethyl ether using a Soxhlet apparatus.

**Lignin.** Lignin was gravimetrically determined as Klason lignin (Femenia, Sánchez, Simal, & Rosselló, 1998b). AIRs were dispersed in 12 M H<sub>2</sub>SO<sub>4</sub> at room temperature for 3 h and then diluted to 1 M H<sub>2</sub>SO<sub>4</sub> and heated at 100 °C for 2.5 h. The insoluble material was recovered by filtration (sinter no. 2) washed thoroughly with hot water (90 °C) until acid free, and dried overnight at 105 °C. The residue weight was recorded as Klason lignin.

**Starch.** The occurrence of starch in the dehydrated samples was tested for by staining the AIRs with an I<sub>2</sub>/KI solution and examining by light microscopy.

**Analysis of carbohydrate composition.** Carbohydrate analysis was performed as described in Femenia et al. (1998b) for neutral sugars from AIRs. Sugars were released from polysaccharides by acid hydrolysis. AIRs samples from both by-products (≈5 mg) were dispersed in 12 M H<sub>2</sub>SO<sub>4</sub> for 3 h followed by dilution to 1 M and hydrolyzed at 100 °C for 2.5 h. A 1 M H<sub>2</sub>SO<sub>4</sub> hydrolysis (100 °C, 2.5 h) was also included to estimate the cellulose content by difference. Neutral sugars were after derivatisation determined as alditol acetates and isothermally separated by GLC at 220 °C on 3% OV225 Chromosorb WHP 100/120 mesh column with Ar as the carrier gas flowing at 20 ml min<sup>-1</sup>. Injector and FID detector temperatures were 230 °C and 240 °C, respectively. Uronic acids were determined by colorimetry as total uronic acid using a sample hydrolyzed for 1 h at 100 °C in 1 M H<sub>2</sub>SO<sub>4</sub>.

### 2.6. Functional properties

Functional properties (FP) measured included swelling (SW), water retention capacity (WRC) and fat adsorption capacity (FAC) (Femenia et al., 1997). FP were measured for all AIRs from peel and pulp samples. For all FP measurements AIRs were grinded to a particle size of 0.180 mm.

**Swelling.** SW was determined using a settled bed volume to measure swelling after equilibration in an excess of solvent. AIRs from dehydrated samples (±0.5 g) were hydrated in 20 mL of distilled water. After equilibration (16 h), the volume of the sample was recorded and expressed as mL water/g AIR.

**Water retention capacity.** WRC was measured after centrifugation of the water insoluble residues. AIR samples (±0.5 g) were hydrated in excess (24 h) in a 20 mL tube, prior to centrifugation at 2000g for 25 min. Excess supernatant was decanted. Water retention was recorded as g water/g AIR.

**Fat adsorption capacity.** FAC was measured as the oil retention capacity. AIR samples ( $\pm 0.5$  g) were mixed with sunflower oil (10 mL), centrifuged at 2000g for 20 min and the excess supernatant was decanted. FAC was expressed as g oil/g AIR.

### 2.7. Solubility

Solubility was measured in conjunction with WRC, as % loss in the original sample dry weight after recovery of insoluble material used to determine WRC (Femenia et al., 1997).

### 2.8. Total polyphenols and antioxidant activity

The total soluble polyphenols were extracted from freeze dried samples of peel and pulp of the canoneta variety sequentially with methanol: water (50/50, v/v) and acetone: water (70/30, v/v) at room temperature for 60 min in each case. The supernatants were combined, concentrated at 40 °C and lyophilized.

Total soluble polyphenols (TP) were spectrophotometrically determined in the polyphenolic extracts by reading absorbance at 765 nm (Folin–Ciocalteu method), using gallic acid as standard and expressing the results as gallic acid equivalents (GAE). The estimation of total phenolics in both extracts was carried out in triplicate.

A Methron 679 Rancimat instrument was used to assess the antioxidant activity of the dehydrated orange by-products. Samples of peel and pulp dehydrated at different temperature were milled and passed through a 0.180 mm aperture sieve. Freeze-dried samples were taken as a reference in both cases. The oxidation took place on commercial sunflower oil, to which samples were added. Approximately, 6 mg of dehydrated sample were suspended directly into 5 g of sunflower oil in a glass cylinder and stirred. The samples were tested by air bubbling through the mixtures in the Rancimat apparatus at 110 °C and at a flow rate of 20 l/h. In order to control the experiment, commercial sunflower oil was used under the same conditions as described above. The antioxidant activity was determined as a protection factor for the rancidity (PF), calculating the ratio between the induction time of the sample and the induction time of the control. Generally, a PF equal to 1 means that the sample has no apparent antioxidant activity, whereas a PF higher than 1 indicates that certain antioxidant activity is exhibited by the sample. A PF less than 1 reveals a pro-oxidant activity (Beddows, Jagait, & Kelly, 2001; Louli et al., 2004). All these measurements were carried out in triplicate.

### 2.9. Statistical analysis

The variability in the physico-chemical data was analyzed by using SPSS version 10.0. Statistical analysis was performed by ANOVA to lead to end the statistical comparisons between groups by using the general linear models

procedure of the statistical package for social science (SPSS, 1997).

## 3. Results and discussion

The initial moisture content of the fresh orange peel and pulp was 78.6 g H<sub>2</sub>O/100 g fresh peel and 84.8 g H<sub>2</sub>O/100 g fresh pulp, respectively. *Citrus aurantium* v. Canoneta has a relatively high percentage of skin, about 39% (w/w) of the fresh orange; whereas the pulp remaining after juice extraction represented around 34% (w/w) on a fresh weight basis (see Table 1). Orange peel and pulp samples were dried until 0.12 g H<sub>2</sub>O/g DM and 0.11 g H<sub>2</sub>O/g DM, respectively. This implied a final water activity of ca. 0.35 for both sample types.

### 3.1. Drying procedure

Both orange by-products were dehydrated at 30 °C, 40 °C, 50 °C, 60 °C, 70 °C, 80 °C and 90 °C. The drying kinetics of orange peel and pulp samples are shown in Fig. 1a and b, respectively. As it can be observed, air-drying temperature had an important influence on the drying rate. As expected, longer drying periods were required at lower drying air temperatures, whereas higher temperatures promoted shorter drying times. For example, at 30 °C, over than 8.3 h were needed to reach a moisture content of 0.12 g H<sub>2</sub>O/g DM in both peel and pulp samples, whereas the same water content could be reached in 1.75 h and 2.5 h for peel and pulp samples, respectively, with an air-drying temperature of 70 °C. In all cases, pulp samples presented relatively longer drying times than peel ones when drying was carried out at the same temperature. Interestingly, pulp samples exhibited similar drying rates when the drying process was performed above 80 °C. Thus, the drying kinetics of 80 °C and 90 °C were practically overlapping, suggesting that over this temperature no significant increase in the drying rate would probably be observed. This phenomenon is known as case-hardening effect. This effect hinders the water release and slows down the drying rate; consequently, drying performed at higher temperature does not promote any further increase in the drying rate (Femenia et al., 2003). Different authors had observed the case-hardening phenomenon during the dehydration of different food products (Demirel & Turhan, 2003; Simal, Femenia, Llull, & Rosselló, 2000).

### 3.2. Color of powdered fibre concentrates

Color is one of the more important quality parameters in dehydrated fruits and vegetables. Undoubtedly, possible color changes would influence the organoleptic properties of dried orange peel and pulp samples and would limit their potential applications (Femenia et al., 2003). Limitations with respect to the use of apple pomace concentrates in very light-colored foods have been reported due to the slightly brown hue of apple pectins caused by enzyme

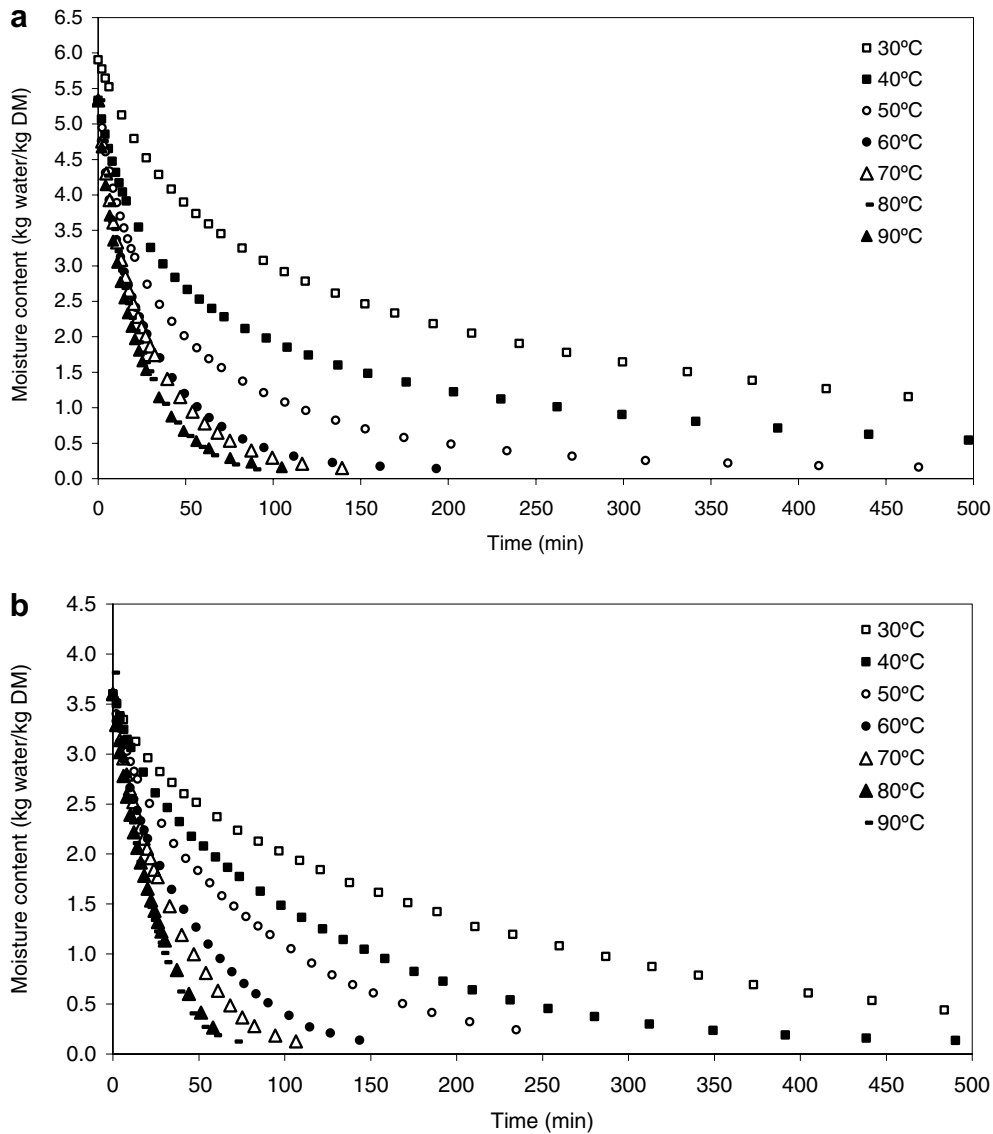


Fig. 1. Drying kinetics corresponding to the dehydration of orange peel (a) and orange pulp (b) samples.

browning (Renard et al., 1997). The Maillard reaction often occurs when foods are heat-treated. Parameters affecting the Maillard and non-enzymic reactions are primarily temperature and the duration of the heat treatment (Chua, Mujumdar, Hawlader, Chou, & Ho, 2001).

In this study, CIELab\* values of samples of both by-products dried at different air-drying temperatures were measured (see Table 2). In addition, a freeze dried sample was taken as a reference for each by-product.

The influence of the air-drying temperature on browning development, taking into account the  $\Delta E$  parameter, did not equally affect samples from both by-products. Thus, orange pulp samples underwent a relatively higher color modification than peel samples. In general, the samples of both by products dehydrated at elevated temperature, i.e. from 70 °C to 90 °C, exhibited relatively high  $\Delta E$  and  $\Delta L$  values. However, the highest browning development was observed for pulp dried at 30 °C, which required an

extended drying period to achieve the final moisture. In particular, the occurrence of a relatively important amount of remaining reducing sugars in this sample could also be responsible for the enzymic browning detected.

### 3.3. Alcohol insoluble residues (AIRs)

The AIR contents of fresh and dehydrated peel and pulp samples, are shown in Fig. 2. In general, both orange by-products presented relatively important amounts of AIR, being the orange peel the part of the fruit which exhibited the largest AIR content. Thus, AIR values ranged from 41.1 to 48.3 g AIR/100 g DM, and from 27.9 to 39.9 g AIR/100 g DM for peel and pulp samples, respectively. The highest values in both samples corresponded to the fresh samples.

Thus, air-drying temperature had an important influence on the percentage of AIR obtained, in particular for

Table 2  
CIELab\* coordinates of orange peel and pulp samples dehydrated at different temperature

	<i>L</i>	<i>a</i> *	<i>b</i> *	$\Delta L$	$\Delta E$
Peel <i>T</i> (°C) <sup>a</sup>					
30	79.36	-1.76	64.09	-6.66	7.170
40	79.64	-2.44	62.13	-6.38	6.959
50	80.11	-0.92	66.69	-5.91	7.348
60	79.90	-1.40	61.61	-6.12	7.258
70	79.00	-0.51	60.51	-7.02	8.793
80	78.56	-0.28	60.09	-7.46	9.419
90	79.60	-1.55	58.01	-6.42	9.290
Fresh	86.02	-4.42	64.08	-	-
Pulp <i>T</i> (°C)					
30	74.85	3.63	45.17	-11.45	14.114
40	77.89	0.79	40.42	-8.41	9.017
50	81.14	0.36	43.13	-5.16	6.883
60	79.38	1.59	42.24	-6.92	8.422
70	78.09	1.51	41.63	-8.21	9.316
80	78.69	2.30	44.21	-7.61	10.095
90	77.30	2.26	43.89	-9.00	11.031
Fresh	86.3	-2.31	39.44	-	-

<sup>a</sup> Freeze dried sample of each by-product was used as a reference.

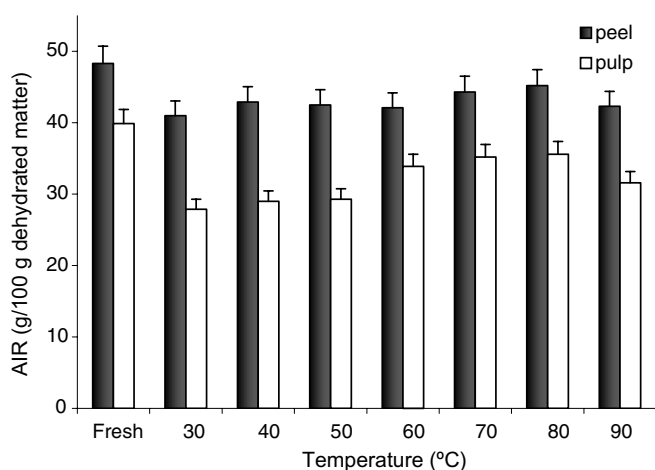


Fig. 2. AIR content of fresh and processed orange peel and pulp samples. (Results are expressed as g of AIR/100 g of dehydrated material).

pulp samples. Thus, peel samples exhibited a similar AIR content for all the different drying treatments applied. For dried pulp samples, the maximum AIR values were obtained between 60 °C and 80 °C, whereas significant lower AIR recoveries ( $p < 0.05$ ) were obtained either at lower or higher temperatures.

### 3.4. Overall composition of AIRs

The moisture, protein, lignin, ash and total carbohydrate of the AIRs from peel and pulp samples were determined. No significant differences ( $p > 0.05$ ) in the overall AIR composition were found between the different orange by-products. Carbohydrate accounted for most of the material recovered in the AIR, representing approximately around 80% of the AIR in both by-products. The relatively

low content of lignin, about 1.2–1.4% in peel and 0.9–1.0% in pulp samples, indicated the absence of secondary walls in both orange tissues. Accounting for the term of dietary fibre (DF) as the sum of cell wall carbohydrates and lignin (Englyst & Hudson, 1996), the DF content of both by-products ranged from 33.1% to 36.5% DM and from 22.6% to 28.3% DM for peel and pulp, respectively. These values are lower than those presented by Larrauri, Rupérez, Bravo, and Saura-Calixto (1996) who reported a total DF content of 69.1% DM in orange skin, and also to those presented by Chau and Huang (2003), of 51.5% DM, but more similar to the value presented by Chang, Lee, Lin, and Chen (1998) of 38.7% DM of the whole orange. These differences could be due to the different varieties of oranges analyzed. Nevertheless, dried orange by-products of the “canoneta” variety have an important DF content in comparison with other dried fruits: 22.1% DM in green apple, 19.3% DM in melon skin or 26.3% DM in peach (Chang et al., 1998).

### 3.5. Carbohydrate composition

Sugars in the AIRs were released using two hydrolytic procedures in order to distinguish the sugars from noncellulosic polysaccharides and cellulose. All AIR preparations were shown to be free of starch, by I<sub>2</sub>/KI staining and also by the low recovery of glucose following hydrolysis in 1 M sulfuric acid. The results, expressed on orange fresh weight basis in order to allow better comparison, are shown in Table 3.

### 3.6. Cell wall polysaccharides

Pectic substances were the predominant type of polysaccharide identified for the cell walls of orange peel and pulp concentrates. In general, both by-products exhibited a rather similar cell wall composition, although AIRs from peel samples contained a slightly higher proportion of pectic substances. The presence of pectic polysaccharides was inferred from the relatively large amounts of uronic acids, arabinose and galactose, and to a minor extent from the occurrence of rhamnose, which could be detected. Interestingly, pectic polysaccharides from pulp samples contained a higher ratio of Ara+Gal/Uronic acids than those identified in peel AIRs, suggesting that pectic polymers from the latter by-product presented a higher degree of branching which could affect to the functional properties of these by-products.

Cellulose was the second cell wall polymer-type in abundance. The occurrence of important amounts of cellulose was inferred from the fact that the bulk of glucose could only be released after Saeman hydrolysis (Auffert, Guillon, Barry, & Thibault, 1994).

In addition, the presence of xylosyl, fucosyl and mannosyl residues in both by-products was indicative of the occurrence of a relatively important amount of hemicellulosic polysaccharides, most probably xyloglucans.

Table 3  
Carbohydrate analysis of fresh and dehydrated orange skin and orange pulp samples (g/100 g orange fresh weight)

Sugar	Drying temperature (°C)							
	Fresh	30	40	50	60	70	80	90
<i>Peel</i>								
Rha	0.12 ± 0.01	0.08 ± 0.01	0.07 ± 0.00	0.10 ± 0.01	0.08 ± 0.00	0.08 ± 0.00	0.08 ± 0.01	0.08 ± 0.01
Fuc	0.03 ± 0.00	0.03 ± 0.00	0.02 ± 0.00	0.03 ± 0.00	0.03 ± 0.00	0.02 ± 0.00	0.03 ± 0.00	0.03 ± 0.00
Ara	0.55 ± 0.01	0.42 ± 0.02	0.54 ± 0.02	0.54 ± 0.02	0.53 ± 0.03	0.54 ± 0.03	0.53 ± 0.03	0.49 ± 0.03
Xyl	0.16 ± 0.01	0.15 ± 0.01	0.16 ± 0.01	0.14 ± 0.01	0.14 ± 0.01	0.16 ± 0.01	0.15 ± 0.01	0.15 ± 0.01
Man	0.17 ± 0.01	0.16 ± 0.01	0.14 ± 0.01	0.17 ± .01	0.15 ± 0.01	0.14 ± 0.01	0.17 ± 0.01	0.16 ± 0.01
Gal	0.55 ± 0.02	0.43 ± 0.02	0.45 ± 0.02	0.49 ± 0.03	0.53 ± 0.03	0.52 ± 0.02	0.53 ± 0.03	0.50 ± 0.02
Glc	1.31 ± 0.05	1.15 ± 0.06	1.20 ± 0.05	1.23 ± 0.07	1.28 ± 0.07	1.29 ± 0.07	1.29 ± 0.06	1.17 ± 0.06
Glc (1 M)	(0.12 ± 0.01)	(0.08 ± 0.01)	(0.11 ± 0.01)	(0.11 ± 0.01)	(0.10 ± 0.01)	(0.10 ± 0.01)	(0.11 ± 0.01)	(0.06 ± 0.01)
Uronic	0.76 ± 0.03	0.65 ± 0.04	0.72 ± 0.03	0.75 ± 0.04	0.73 ± 0.04	0.74 ± 0.04	0.65 ± 0.03	0.61 ± 0.03
Total	3.65 ± 0.08	3.07 ± 0.11	3.30 ± 0.08	3.45 ± 0.09	3.46 ± 0.07	3.49 ± 0.09	3.42 ± 0.05	3.19 ± 0.08
<i>Pulp</i>								
Rha	0.09 ± 0.01	0.08 ± 0.01	0.07 ± 0.01	0.09 ± 0.01	0.08 ± 0.01	0.07 ± 0.01	0.06 ± 0.01	0.06 ± 0.01
Fuc	0.03 ± 0.00	0.02 ± 0.00	0.02 ± 0.01	0.01 ± 0.00	0.02 ± 0.00	0.02 ± 0.00	0.02 ± 0.00	0.03 ± 0.00
Ara	0.21 ± 0.02	0.13 ± 0.01	0.15 ± 0.02	0.17 ± 0.02	0.19 ± 0.03	0.19 ± 0.02	0.20 ± 0.02	0.19 ± 0.02
Xyl	0.09 ± 0.01	0.09 ± 0.01	0.07 ± 0.01	0.06 ± 0.01	0.06 ± 0.01	0.08 ± 0.01	0.07 ± 0.01	0.07 ± 0.01
Man	0.11 ± 0.01	0.08 ± 0.01	0.10 ± 0.01	0.07 ± 0.01	0.08 ± 0.01	0.09 ± 0.01	0.09 ± 0.01	0.08 ± 0.01
Gal	0.28 ± 0.03	0.22 ± 0.02	0.24 ± 0.03	0.24 ± 0.03	0.28 ± 0.04	0.27 ± 0.03	0.26 ± 0.02	0.22 ± 0.02
Glc	0.59 ± 0.05	0.48 ± 0.04	0.53 ± 0.05	0.56 ± 0.06	0.58 ± 0.08	0.58 ± 0.06	0.58 ± 0.06	0.56 ± 0.06
Glc (1 M)	(0.03 ± 0.00)	(0.04 ± 0.00)	(0.03 ± 0.00)	(0.03 ± 0.01)	(0.02 ± 0.01)	(0.03 ± 0.00)	(0.02 ± 0.00)	(0.03 ± 0.01)
Uronic	0.32 ± 0.02	0.19 ± 0.03	0.25 ± 0.02	0.36 ± 0.03	0.30 ± 0.04	0.33 ± 0.03	0.27 ± 0.02	0.18 ± 0.01
Total	1.72 ± 0.04	1.30 ± 0.03	1.43 ± 0.07	1.46 ± 0.08	1.60 ± 0.07	1.64 ± 0.05	1.55 ± 0.07	1.39 ± 0.04

These results are in broad agreement with the results of Chang et al. (1998) reporting a large amount of uronic acids, galactose, arabinose and glucose as the main sugars for the cell walls of sweet orange. However, they differ from the results presented by Auffert et al. (1994) who reported higher amounts of xylose than glucose. This fact could probably be due to the different varieties of oranges studied. For orange peel tissues, Chau and Huang (2003) reported similar results than these obtained in this work; being arabinose, galactose, glucose and uronic acids the main cell wall sugars detected. Englyst, Bingham, Runswick, Collinson, and Cummings (1988) reported a larger amount of uronic acids for orange flesh, although the analyses were performed on whole orange flesh samples before juice extraction. In the same study, pectic polymers, rich in uronic acid were the main cell wall components present in orange juice.

In general, the major losses of cell wall sugars, for both type of orange by-products, were detected when the drying procedures were carried out at high temperature (i.e. 90 °C). However, drying at low temperature (i.e. 30 °C), requiring longer time of sample exposure to the heat, also promoted important losses of pectic polymers. This could be inferred from the lower amounts of uronic acids recovered, and also of arabinose and galactose units. Pectic substances are more susceptible to enzyme or heat induced chemical degradation than other polysaccharide components of the cell wall (Levi, Ben-Shalom, Plat, & Reid, 1988).

The significant lower recoveries of cellulosic glucose in several samples might also suggest some major disruption of the cell wall arrangement during processing, despite cellulose being the most resistant type of cell wall polymer.

Samples of orange peel dried at 50 °C, 60 °C and 70 °C did not exhibit significant differences for the cell wall composition, and were similar to the fresh sample. Further, fresh pulp samples and those dried between 60 °C and 80 °C also exhibited minor differences in their carbohydrate composition.

### 3.7. Functional properties

Functional properties are related to the chemical structure of the plant polysaccharides. Therefore, the drying process may alter the physico-chemical properties of the original products, modifying their functional properties (Femenia, Bestard, Sanjuan, Rosselló, & Mulet, 2000). Therefore, in order to evaluate possible modifications affecting the structural arrangement of cell wall polysaccharides from peel and pulp samples, hydration-related properties such as swelling (SW) and water retention capacity (WRC), and the fat adsorption capacity (FAC) were measured on fresh and processed AIRs. The results are shown in Fig. 3a (SW), b (WRC) and c (FAC).

*Swelling.* Pulp AIRs exhibited higher average swelling figures than peel AIRs for fresh samples and also for the whole range of air-drying temperatures considered. Further, SW values for peel AIRs seemed not to be affected by the drying temperature, whereas SW values for pulp AIRs were clearly influenced by the air drying temperature (see Fig. 3a). For example, SW figures decreased from 29.4 mL H<sub>2</sub>O/g AIR to 23.6 mL H<sub>2</sub>O/g AIR for samples dried at 80 and 90 °C, respectively, which represents a decrease of ca. 20%. The highest SW value was obtained for pulp fibre dried at 40 °C.

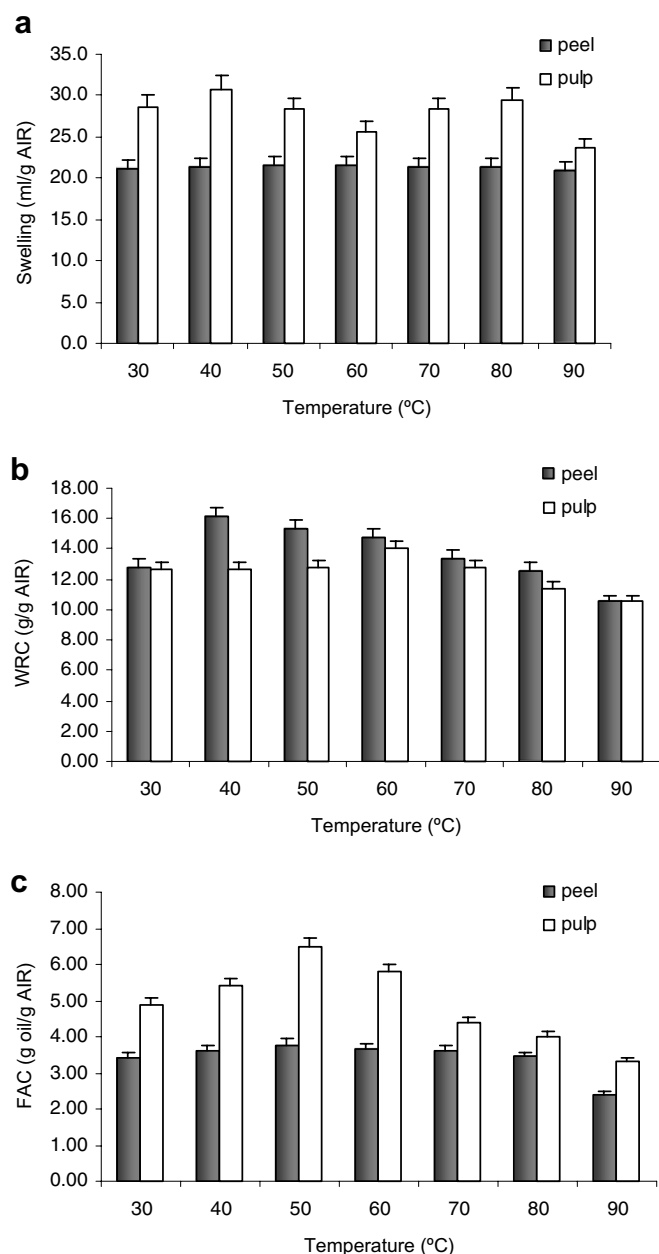


Fig. 3. Functional properties of AIRs from fresh and dehydrated orange peel and pulp tissues. (a) Swelling; (b) WRC; (c) FAC.

**Water retention capacity.** WRC values measured for peel AIRs were higher than those obtained for pulp samples dried at the same temperature. Differences were particularly significant ( $p < 0.01$ ) for fresh peel sample and those dried at 40 °C and 50 °C in comparison to the remaining processed samples. Despite these differences, from 60 °C to 90 °C, both by-products exhibited a similar trend. Thus, an increase in the air-drying temperature promoted a clear decrease in the WRC values (see Fig. 3b).

WRC values ranged from 10.5 to 16.1 g H<sub>2</sub>O/g AIR and from 10.6 to 14.0 g H<sub>2</sub>O/g AIR for orange peel and pulp, respectively. These values are similar to those measured by Adams, Evans, Oakenfull, and Sidhu (1986) for orange processing by-products, and higher than the WRC of most

of agricultural by-products reviewed by Grigelmo-Miguel and Martín-Belloso (1999).

**Fat adsorption capacity.** In general, dehydration promoted a general decrease of the FAC of all processed samples in comparison to the FAC corresponding to the fresh samples (see Fig. 3c). Moreover, pulp AIRs exhibited significantly higher FAC values than peel AIRs within the range of temperatures studied and also in the case of the fresh samples. In addition, drying temperature promoted important changes in the FAC values of the different samples, in particular for the pulp AIRs. A gradual increase from 30 to 50 °C was observed followed by a significant decrease from 60 to 90 °C. To a minor extent, a similar trend could be observed for peel samples.

Nevertheless, FAC values, either for processed peel or pulp AIRs, were higher than other FAC values reported for the DF concentrates from different orange varieties, from 0.8 to 1.3 g oil/g fibre concentrate (Grigelmo-Miguel & Martín-Belloso, 1999).

### 3.8. Solubility

DF is often classified as soluble DF and insoluble DF (Gorinstein et al., 2001). Because the term solubility refers simply to fibres that are dispersible in water, the term is somewhat inaccurate (Figueroa, Hurtado, Estévez, Chiffelle, & Asenjo, 2005). However, the soluble–insoluble ratio is important for both dietary/functional properties derived from DF.

Significant differences in solubility, depending on the dehydration temperature, were observed for both types of orange by-products (Fig. 4). Solubility values ranged from 27.3% to 37.8% AIR and from 25.9% to 38.5% AIR for peel and pulp samples, respectively. In both orange by-products, the lowest solubility values were measured for samples dried at 90 °C, whereas samples dried within the range from 40 °C to 70 °C exhibited significant higher values ( $p < 0.01$ ), being similar to the solubility determined for

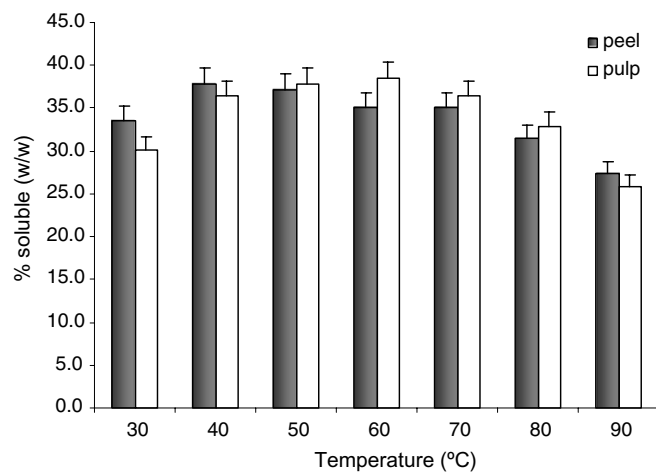


Fig. 4. Solubility of AIRs from fresh and processed orange peel and pulp samples.



the fresh samples. The low solubility values detected for samples dried at 30 °C could be attributed to the longer exposure to the heat, required to achieve the final moisture content.

The ratios soluble–insoluble DF, calculated for the peel and pulp AIRs, were close to 1:2 in most cases. In fact, it is generally accepted that those fibre sources suitable as food ingredient should have a soluble–insoluble ratio of approximately 1:2 (Schneeman, 1987). Only for samples dried at higher temperatures, 80 °C and 90 °C, and also, in the case of pulp, the sample dried at 30 °C, presented a higher proportion of insoluble matter. The decrease in solubility could not only be due to the degradation of pectic substances during processing but also caused by structural modifications affecting to these polymers during the removal of water.

### 3.9. Antioxidant activity

Table 4 shows the content of the total soluble polyphenols (TP) of fresh “canoneta” orange peel and pulp samples together with values from other citrus fruit products reported in the literature.

As it can be observed, “canoneta” peel sample contained a significantly higher amount of TP than pulp sample. A similar observation has been reported by different authors in the case of different citrus by-products (Gorinstein et al., 2001). Further, peels of several other fruits such as

Table 4  
Total extractable polyphenols in peel and/or pulp from different citrus by-products (results are given as mg GAE/100 g DM)

Citrus fruit	Peel	Pulp
Orange ( <i>C. aurantium</i> cv. Canoneta) <sup>a</sup>	0.51 ± 0.03	0.42 ± 0.02
Orange ( <i>C. sinensis</i> cv. Navel) <sup>a</sup>	0.37 ± 0.03	–
Lemon ( <i>C. limon</i> cv. Yenben) <sup>a</sup>	0.59 ± 0.02	–
Lemon ( <i>C. limon</i> cv. Meyer) <sup>a</sup>	0.81 ± 0.08	–
Grapefruit ( <i>C. paradisi</i> ) <sup>a</sup>	0.30 ± 0.02	–
Mandarine ( <i>C. reticulata</i> cv. Ellendale) <sup>a</sup>	0.61 ± 0.08	–
Orange ( <i>C. sinensis</i> ) <sup>b</sup>	0.89 ± 0.05	0.77 ± 0.05
Lemon ( <i>C. limon</i> ) <sup>b</sup>	0.95 ± 0.05	0.82 ± 0.05
Grapefruit ( <i>C. paradisi</i> ) <sup>b</sup>	0.78 ± 0.05	0.68 ± 0.05
Orange ( <i>C. sinensis</i> cv. Valencia) <sup>c</sup>	0.16	–
Lime ( <i>C. aurantifolia</i> cv. Persa) <sup>c</sup>	0.35	–
Orange ( <i>C. sinensis</i> cv. Navel) <sup>d</sup>	0.01–0.25	–
Buntan ( <i>C. grandis</i> Osbeck) <sup>e</sup> flavedo	0.11	–
Buntan ( <i>C. grandis</i> Osbeck) <sup>e</sup> albedo	0.09	–
Citron ( <i>C. medica</i> ) <sup>f</sup>	–	0.20
Blood red orange ( <i>C. sinensis</i> ) <sup>f</sup>	–	0.40
Orange ( <i>C. sinensis</i> ) <sup>g</sup>	0.43	–
Grapefruit ( <i>C. paradisi</i> ) <sup>g</sup>	0.76	–
Tangerine ( <i>C. reticulata</i> ) <sup>g</sup>	0.51	–

<sup>a</sup> Li et al. (2006).

<sup>b</sup> Gorinstein et al. (2001).

<sup>c</sup> Larrauri et al. (1996).

<sup>d</sup> Anagnostopoulou et al. (2006).

<sup>e</sup> Mokbel and Hashinaga (2006).

<sup>f</sup> Jayaprakasha and Patil (2007) (as catechin equivalents).

<sup>g</sup> Alicia et al. (2005).

\* Experimental results are expressed as the means ( $n = 3$ ) ± SD.

apple, pear, pomegranate, mango and peach, have also been found to contain higher amount of phenolics than the edible fleshy parts (Berardini, Knödler, Schieber, & Carle, 2005; Gorinstein et al., 2002; Li et al., 2006).

The variability of the results in the TP content of different citrus fruits observed in Table 4 can be attributed not only to the different fruit varieties but also to the different solvents used in the extraction process.

Drying processes, and in particular high temperatures and long drying times, might destroy some of the phenol compounds (Li, Smith, & Hossain, 2006). In the dried materials, all the plant cell components adhere together in the absence of water, and possibly making the extraction with solvent more difficult; as a result, the overall recoveries might be lower than expected.

In this study, the effect of the dehydration temperature on the antioxidant activity was measured using the Rancimat method for all the dried samples from both orange by-products. The results of the protection factor (PF) for rancidity are shown in Fig. 5.

All dried samples presented PF values higher than 1, indicating a certain antioxidant capacity. However, peel samples exhibited significant higher values ( $p < 0.01$ ) than pulp samples for PF within the whole range of temperatures studied. Thus, the average PF value for peel samples was 1.52, which compared to the average PF value of pulp, approximately 1.37, represents a difference in the antioxidant capacity of ca. 10%.

The air-drying temperature affected the PF values of both sample types, and this fact was clearer on dried pulp samples. The highest antioxidant capacity either for peel or pulp was determined when dehydration took place at 60 °C. Drying at higher temperatures (i.e. 80 and 90 °C) or at temperatures which implied longer drying times (i.e. 30 and 40 °C) promoted a decrease of the antioxidant capacity. This effect was especially significant for pulp samples.

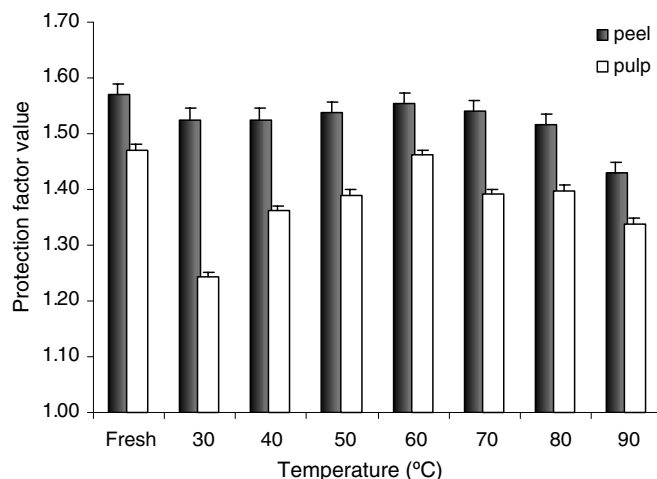


Fig. 5. Antioxidant capacity of fresh and dehydrated orange peel and pulp samples.

According to these results, it would seem clear that the most appropriate drying temperature in order to preserve the antioxidant capacity of both by-products would be around 60 °C, suggesting that antioxidant compounds from peel samples have a higher resistance to heat degradation. The flavonoid content of several fractions and residues of extracts of sweet orange peel has been recently qualitatively and quantitatively determined by HPLC-diode array detection-electrospray ionization mass spectrometry (Anagnostopoulou, Kefalas, Kokkalou, Assimopoulou, & Papageorgiou, 2005). The effects of air-drying temperature on flavonoids and other phenolic constituents are underway.

#### 4. Conclusions

The results of this study suggest that dehydrated citrus by-products obtained from orange peel and the remaining pulp after juice extraction could be suitable sources of DF and antioxidants. In fact, dried orange peel samples contained larger amounts of DF, and, also, exhibited higher antioxidant capacity. In general, the by-products studied proved to be quite resistant to the different heat treatments applied within the range from 40 °C to 70 °C. Further, both types of samples showed appropriate ratios of soluble–insoluble DF to be used as fibre supplements.

However, the study suggests that processing history of DF concentrates with associated bioactive compounds should be taken into consideration in order to obtain a better assessment before these concentrates could be incorporated as ingredients in a large variety of food products (functional foods), or even to be used as pharmaceutical supplements (Femenia, 2007).

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